UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

US ENDODONTICS, LLC,
Petitioner,

v.

GOLD STANDARD INSTRUMENTS, LLC,
Patent Owner.

Case IPR2015-00632
Patent 8,727,773 B2

DECLARATION OF ROBERT SINCLAIR, PH.D.

I, Robert Sinclair, Ph.D., hereby declare and state that:

1. I make the following declaration based on my knowledge and belief.

Education and Professional Background

2. I received a B.A. in Materials Science in 1968 and a Ph.D. in Materials Science in 1972, both from Cambridge University. After receiving my Ph.D., I worked from 1973-1977 as a Postdoctoral Research Engineer at the University of California, Berkeley.

3. Since 1977, I have been employed at Stanford University in Stanford, California, where I have successively served as Assistant Professor in Materials Science and Engineering (1977-1980), Associate Professor with tenure in Materials Science and Engineering (1980-1984) and Professor of Materials Science and Engineering (1984 to Present). In 2009, I was appointed the Charles M. Pigott Professor in the School of Engineering at Stanford University.

4. From 2004-2014, I served as the chair of the Materials Science and Engineering Department at Stanford University. From 2002 to 2013, I was the Director of the Stanford Nanocharacterization Laboratory, and from 2010-2012, I was the Director of the Bing Overseas Studies Program at Stanford University. I have had a number of appointments as a Visiting Professor at institutions around the world, including the HREM Laboratory at Cambridge University in the United Kingdom and Matsushita Electric Industrial Company in Japan.

5. I have authored more than 240 scientific research papers, published over 200 articles at national and international scientific meetings, and made over 500 presentations at conferences, university departments, and research laboratories world-wide. My publications, which are listed on my *curriculum vitae* attached hereto as Exhibit A, are in the areas of materials science, and include investigations on the properties of nickel-titanium alloys. I have also authored and/or edited several books and book chapters, and I hold two patents.

6. I have served as a member on the Editorial Board for the Journal of Applied Physics (1994-1996) and the Journal of Electron Microscopy (1996-present), among other journals. I routinely review articles for scholarly journals.

7. I have taught more than 6,000 students in undergraduate and graduate courses, including, among others, Introduction to Materials Science; Imperfections in Crystalline Solids; Atomic Arrangements in Solids; Nanostructure and Characterization; X-ray Diffraction Laboratory; Nano-Characterization of Materials; Transmission Electron Microscopy; and Microscopic World of Technology.

8. My current research interests are in the structure-property-processing correlations in materials, using high-resolution microscopy and diffraction techniques, application to development of integrated circuit and magnetic recording materials and introduction of in situ high resolution electron microscopy.

This includes their application to understanding phase transformations and deformation of nitinol alloys, correlated with Differential Scanning Calorimetry (DSC) analysis.

9. Throughout the course of my career, I have received various honors and awards, as described in detail in my *curriculum vitae*. Some of the awards I have received include the Robert Lansing Hardy Gold Metal from the Metallurgical Society of AIME in 1976, the Alfred P. Sloan Foundation Fellowship in 1979, the Distinguished Scientist Award (Physical Sciences) from the Microscopy Society of America in 2009, and the David M. Turnbull Lectureship Award from the Materials Research Society in 2012.

10. Based on my experience and qualifications, I am qualified to render opinions in the field of nickel-titanium alloys. I am an expert in the field of materials science and engineering, particularly in electron microscopy and material structure and phase transformations, with several well-cited articles on the behavior of nitinol alloys.

My understanding of the Proceeding

11. I have been retained in this matter by Rothwell, Figg, Ernst & Manbeck, P.C. of Washington, D.C., the attorneys representing the Patent Owner in this proceeding. I am being compensated at my regular consulting rate of $600 per hour for time spent consulting, plus expenses.

12. I understand that the real parties in interest for the Patent Owner are Gold Standard Instruments, LLC; Dentsply International Inc.; and Tulsa Dental Products LLC d/b/a Tulsa Dental Specialties.

13. I do not have a financial interest in any of the real parties in interest or in the outcome of this proceeding.

14. My opinions provided in this declaration are as an independent expert witness.

15. Prior to my involvement in this matter, my previous personal contact with the real parties in interest was as an independent expert witness for related litigation in federal district court. That litigation is styled under the caption *Dentsply International, Inc. and Tulsa Dental Products LLC d/b/a Tulsa Dental Specialties v. US Endodontics, LLC,* Case No. 2:14-cv-00196-JRG (E.D. Tenn.).

16. I am informed that an *inter partes* review proceeding involving claims 1-17 of U.S. Patent No. 8,727,773 B2 (the "'773 patent") has been instituted by the Patent Trial and Appeal Board at the United States Patent and Trademark Office.

17. I am informed that the '773 patent issued from U.S. patent application serial no. 13/455,841, filed Apr. 24, 2012, which is a continuation of application serial no. 13/336,579, filed Dec. 23, 2011, which is a continuation of application serial no. 12/977,625, filed Dec. 23, 2010, which is a division of application serial no. 11/628,933, filed Dec. 7, 2006, which is a national stage entry of international

application no. PCT/US05/19947, filed Jun. 7, 2005, which claims benefit of application serial no.60/578,091, filed Jun. 8, 2004.

18. In forming my opinions set forth in this declaration, I relied on my experience, education, knowledge, and the materials discussed or cited in my declaration.

My understanding of the legal standards

19. I have been informed that for a prior art reference to anticipate a patent claim, the reference must disclose all of the limitations, either expressly or inherently, arranged in the same way as in the claim. I have been informed that express anticipation means that each and every limitation of a claim is expressly disclosed in the prior art reference. I have also been informed that inherent anticipation requires that a person of ordinary skill in the art would recognize that a missing descriptive feature of the claim is necessarily present in the prior art reference.

20. I have been informed that a determination of obviousness requires consideration of: (1) the scope and content of the prior art; (2) the differences between the prior art and the claims at issue; (3) the level of ordinary skill in the art; and (4) objective evidence of nonobviousness, if any.

21. I have also been informed that when combining references in an obviousness analysis it is important to identify a reason and rationale that would

have prompted a person of ordinary skill in the art to combine the teachings of different prior art references to achieve the claimed invention. I have been informed that obviousness cannot be based on the hindsight selection of elements of the claimed invention from among the disclosures of prior art references. I have also been informed that a prior art must be considered as a whole, including portions that would teach away from the claimed invention. I have been informed that a reference may teach away from a particular combination when the prior art reference criticizes, discredits, or disparages such a combination, or otherwise suggests taking a path that is divergent from that path leading to the claimed invention. I understand that it is improper to pick and choose from different portions of references.

22. I understand that certain determinations are to be analyzed from the perspective of a "person having ordinary skill in the art," and that such a person would be involved with the technology at issue at the time of the claimed invention. Based on my review of the '773 patent, my review of the materials relied on in the petition for *inter partes* review, and my own research and academic experience, I believe that a person of ordinary skill in the art would have had familiarity with nickel titanium alloys and would likely have at least a B.S. degree in material science, metallurgy, or related field and several years of experience in metallurgy and nickel titanium alloys in particular. Alternatively, a person of

ordinary skill in the art could have a higher level of education and less experience or vice-versa. The relevant experience could be in industry, academia, government, private practice, a clinic or any other setting so as to provide an understanding of the structural or mechanical properties of nickel titanium endodontic instruments.

23. In forming my opinions I considered the level of skill for a person of ordinary skill in the art and the scope and content of the prior art in the time period of around June 8, 2004, the claimed priority date of the '773 patent.

Background relating to nickel-titanium files

24. Special properties of nickel titanium alloys containing an approximately 50:50 atomic ratio of nickel to titanium (known as "nitinol") were first discovered at the Naval Ordnance Laboratory in 1959. Those properties are known as superelasticity and shape memory. Almost thirty years passed before reports of using nitinol in endodontic hand files appeared. *See*, *e.g.*, Walia *et al.*, *An initial investigation of the bending and torsional properties of Nitinol root canal files*. J. Endod. 1988, 14, 346-351 (Ex. 1003).

25. Near equi-atomic nickel-titanium alloys have different crystal structures depending on temperature. At high temperatures, nickel-titanium alloys assume a "cubic" crystal structure (austenite). At low temperature, nickel-titanium alloys spontaneously transform to a more complicated "monoclinic" crystal structure (martensite). A "rhombohedral" crystal structure is sometimes observed between

the austenitic and martensitic phases in some nickel-titanium compositions (R-phase). The reversible transition from austenite to martensite occurs through a shear, martensitic reaction. The martensitic transformation may be temperature-induced or stress-induced.

26. The thermal energy absorbed or given off during the transformation between austenite (cubic), the intermediate R-phase (rhombohedral), and martensite (monoclinic) crystal structures can be measured using differential scanning calorimetry (DSC). It is established in the scientific literature that thermal analysis by DSC is a scientifically accurate and reliable tool for determining the transformation temperatures in nickel titanium alloys.

27. Upon heating from a very low temperature, the crystal structure of nickel titanium transforms from martensite to austenite, meaning that the alloy progresses from about 0% austenite and 100% martensite to about 100% austenite and 0% martensite. The end of the transition—the point at which the material reaches about 100% austenite—is the austenite finish temperature (A_f). For nickel titanium alloys, the important data points on a DSC curve during the heating cycle are the austenite start temperature (A_s) and the austenite finish temperature (A_f). Upon cooling from a high temperature, the crystal structure of nickel titanium transforms from austenite to martensite, and the important DSC data points are the martensite start temperature (M_s) and the martensitic finish temperature (M_f). Additional

DSC data points, known as the R-phase start temperature (R_s) and the R-phase finish temperature (R_f) may be observed during the heating and/or cooling cycles if the alloy transitions through the intermediate R-phase.

28. The applicable ASTM Standard, F2004 -05 (2010) *Standard Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis* ("ASTM Standard") (Ex. 2032), explains how to perform a DSC test, determine the data points of the DSC curve (*i.e.*, A_s, A_f, M_s, M_f), and interpret the data. To determine the data points, section 11.2 states: "Draw the tangents to the cooling and heating spikes through the inflection points as shown in Fig. 1." Ex. 2032 at 2. Section 11.3 states: "Obtain M_s, M_f, A_s, and A_f as the graphical intersection of the baseline with the extension of the line of maximum inclination of the appropriate peak of the curve as shown in Fig. 1." *Id*. Figure 1 is below.

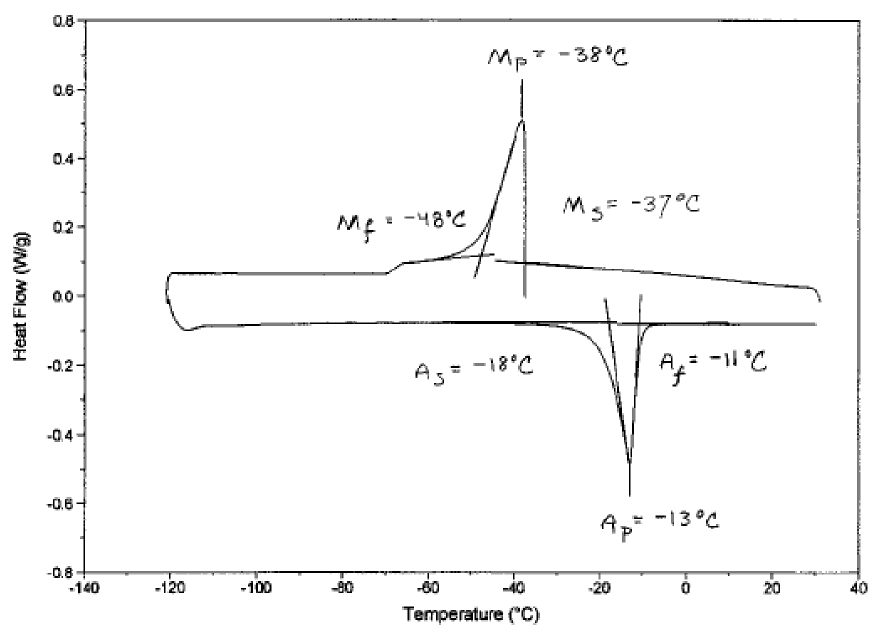


FIG. 1 DSC Curve for Nickel-Titanium (NiTi)

Thus, per the ASTM, M_s and M_f are obtained from the cooling curve (top curve in the above figure) and A_s and A_f are obtained from the heating curve (bottom curve in the above figure). The R_s and R_f temperatures, when present, are typically obtained in the same manner.

29. Superelastic nickel-titanium endodontic rotary files were well-known and popular files prior to the invention of the '773 patent. Ex. 2028, ¶¶ 22-32; Ex. 1002, ¶ 25. The applicable international standard was ISO 3630-1, entitled *Dental Root-Canal Instruments—Part 1: Files, reamers, barbed broaches, rasps, paste carriers, explorers and cotton broaches* ("ISO 3630").[1] Ex. 1016. This first edition of the international standard was published in 1992 before nickel-titanium endodontic files were common and thus only discusses stainless steel endodontic files. *Id*. at 2 (section 4.1.1). ISO 3630 includes information and requirements related to the sizes, materials, and dimensions of endodontic instruments. Ex. 1016 at 1-11. It also includes testing specifications. *Id*. at 12-15.

30. The bending test is in section 6.4. *Id*. at 13-14. The test requires the removal of the handle at the point where the handle is attached to the instrument shaft. *Id*. at 13. The tip of the test piece (file) is then set 3 mm into a chuck on a torque-measuring device such that the test piece is perpendicular to the axis of the

[1] ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies. Ex. 1017 at *iv*.

motor. *Id*. The testing apparatus is set to stop the angular deflection at 45°. *Id*. A catch pin is mounted on the motor shaft of the device, and upon activation of the device, the catch pin will press against the shaft end of the instrument to bend the test piece (file) until it reaches an angular deflection of 45°. *Id*. The force is then recorded. *Id*.

31. One may measure the degree of permanent deformation; *i.e.*, the permanent deformation resulting from the ISO 3630 bend test. Some devices allow one to measure the permanent deformation angle directly from the testing apparatus. Ex. 2029 at 240. Alternatively, a calibrated protractor can be used to determine the degree of permanent deformation after testing in accordance with the ISO 3630 bend test. Ex. 2030 (Ex. A at 2).

The ’773 patent

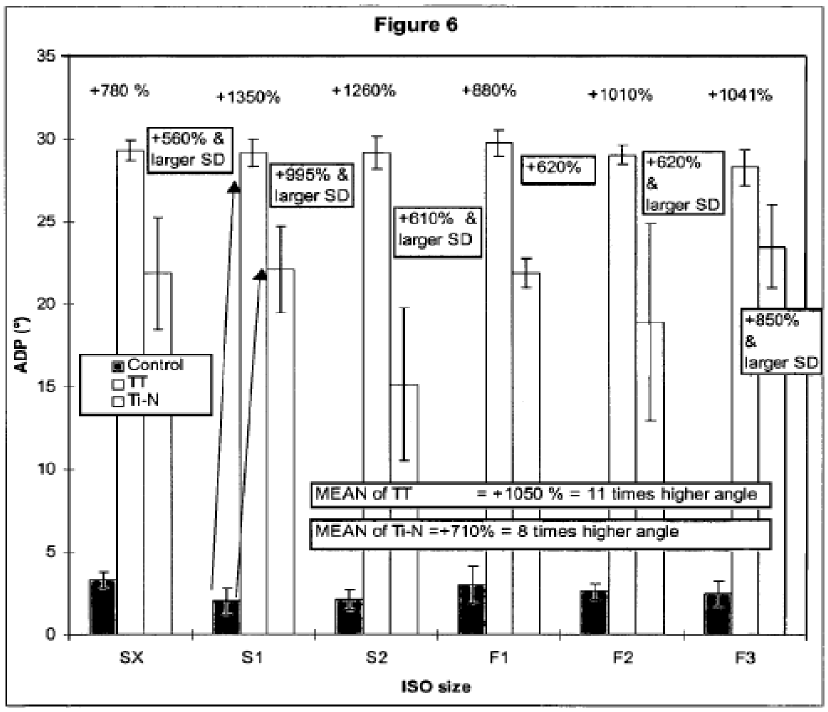
32. The ’773 patent discloses an improved process for manufacturing or modifying dental instruments used in performing root canal therapy on a tooth. Ex. 1001 at 2:56 - 3:16.

33. The ’773 patent discusses problems encountered when performing a root canal therapy on a curved root canal. Ex. 1001 at 2:13-23. For example, stiffer files are difficult to insert through the curved portion of a canal, and in some cases will cut only on the inside of the curve. *Id*.

34. The ’773 patent discusses, in one embodiment, a method of modifying

nickel titanium endodontic files by heat-treating the shank at a temperature above 25°C. Ex. 1001 at 4:12-25. The ’773 patent states that the heat treatment can be conducted at a temperature from 400°C up to but not equal to the melting point of the alloy. *Id*. The ’773 patent states that the heat treatment temperature can be from 475-525°C or 500°C. *Id*. The ’773 patent states that other temperatures are suitable, as they are dependent on the time period selected for heat exposure. *Id*.

35. The ’773 patent includes representative methods of modifying endodontic instruments using heat treatment, and the heat-treated files maintain a deformed shape after bend testing. Ex. 1001 at 8:32-59 (Example 4). Thirty files for each of six different sizes were used to study the angle of permanent deformation after testing in accordance with ISO 3630. The files comprised 54-57 weight percent nickel and 43-46 weight percent titanium. For each of the six different files, ten were heat-treated at 500°C for 75 minutes (TT), ten of each kind were coated with titanium nitride with inherent heat-treatment (Ti-N), and ten of each kind were not heat treated (Control). The results of the bend testing are shown in Figure 6. *Id*. Figure 6 is set out below.



36. The '773 patent shows that TT files maintained about 28-30 degrees of permanent deformation after testing in accordance with ISO 3630. Ex. 1001, Figure 6. The Ti-N coated files maintained about 15-24 degrees of permanent deformation after testing in accordance with ISO 3630. *Id*. The unheated, superelastic Control files maintained about 2-4 degrees of permanent deformation after testing in accordance with ISO 3630. *Id*. Thus, a person of ordinary skill in the art would understand that in this patent a superelastic file is meant to encompass files with a *de minimis* amount of residual bend after the ISO 3630 bend test. A person of ordinary skill in the art also would understand that the claimed invention requires that the heat treated instrument must have significant deformation of greater than 10 degrees after the ISO 3630 bend test.

37. I disagree with Dr. Goldberg's statement that superelastic files must recover 100% of their original shape after undergoing the bend test. This is contrary to the teaching of the '773 patent, as well as my own experience with nickel titanium. During his cross examination, Dr. Goldberg stated that he was applying Dr. Sinclair's definition of superelasticity. Ex. 2034 at 20:8 - 25:8. That is not true. I do not define superelasticity as requiring 100% recovery after deformation.[2] During my deposition, I explained multiple times that superelasticity allows for some residual deformation. Ex. 2037 at 136:8 - 138:3; 143:4-14. Further, when I testified in Court at the hearing regarding a preliminary injunction, I made clear that my understanding of superelasticity, including how that term is used in the context of the '773 patent, allowed for a small amount of residual deformation (*i.e.*, up to ~3 %) after the bend test. Ex. 2001 (Second Substitute) at 294:22 - 297:12.

---

[2] It appears that Petitioner and Dr. Goldberg are attributing this definition to me because they have misinterpreted a general comment I made during my deposition in the related litigation about an object recovering its complete original state (Ex. 2037 at 105:23 - 106:5) and because of a similar statement in my expert report indicating that a superelastic file will return to its "original straight alignment" (Ex. 2038 at ¶ 65). Neither of those statements, however, was meant to exclude a *de minimis* amount of deformation.

38. It is well known in the art that superelastic materials can exhibit residual plastic strain called "permanent set." For example, Pelton Figures 4 and 5 display a number of good examples of permanent set. Ex. 1006 at 111-12. Pelton even refers to the "superelastic flags" and notes that "the permanent set also increases with temperature." *Id*. at 111.

39. The fact that Dr. Goldberg is taking the position that superelasticity requires 100% recovery of a file's original shape makes me doubtful of his knowledge and experience in this area. I am not aware of anyone in the art defining superelasticity as requiring 100% recovery after undergoing a bend test.

40. The '773 patent also discusses other studies that measure torsion (reported in g·cm), maximum torque at 45° of flexion (reported in g·cm), and fatigue (reported in cycles to failure). Ex. 1001 at 7:18-43 (Example 1); 7:45 - 8:2 (Example 2); 8:61 - 9:18 (Example 5). In each study, thirty files for each of six different sizes were used. The files comprised 54-57 weight percent nickel and 43-46 weight percent titanium. For each of the six different files, ten were heat-treated at 500°C for 75 minutes (TT), ten of each kind were coated with titanium nitride with inherent heat-treatment (Ti-N), and ten of each kind were not heat treated (Control). The results of the testing are shown in Figures 3-5 and 7. *Id*.

41. The '773 patent summarizes these studies and states that the files heat-treated at 500°C for 75 minutes exhibited a higher resistance to torsion breakage,

can withstand increased strain, have higher flexibility, have increased fatigue life, and maintain any acquired shape upon fracture better when compared to untreated files. Ex. 1001 at 9:19-23. The '773 patent states that the files prepared according to the invention overcome the problems encountered when cleaning and enlarging curved root canals during root canal therapy. Ex. 1001 at 9:23-30.

42. The '773 patent claims are directed to methods for manufacturing or modifying an endodontic instrument for use in performing root canal therapy on a tooth. Ex. 1001 at 9:43 - 10:57. Each of the methods requires: (a) providing an elongate shank having a cutting edge extending from a distal end of the shank along an axial length of the shank, the shank comprising a superelastic nickel titanium alloy; and (b) after step (a), heat-treating the entire shank at a temperature from 400°C up to but not equal to the melting point of the superelastic nickel titanium alloy, wherein the heat treated shank has an angle greater than 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO Standard 3630.1. *Id.*

43. The '773 patent claims, therefore, require that a superelastic nickel titanium shank be heat treated over 400°C, and that the resulting shank must maintain an angle greater than 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO 3630.

44. I disagree with Dr. Goldberg's statement that the "wherein" clause in

the ’773 patent claims (“wherein the heat treated shank has an angle greater than 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO Standard 3630.1”) can be satisfied if the prior art teaches heat-treated nickel titanium endodontic instruments that are permanently deformable or have austenite finish temperatures above body temperature.

Ex. 1002 at 39.

45. First, the claims require a particular amount of permanent deformation: an angle greater than 10 degrees of permanent deformation after torque at 45 degrees of flexion. As noted above, a *de minimis* amount of permanent set is observed when superelastic endodontic files are subjected to the ISO 3630 bend test. Accordingly, a finding that “endodontic instruments are permanently deformable” is not sufficient to satisfy the claim limitation.

46. Second, the claims do not refer to austenite finish temperatures. The ’773 patent does not mention austenite finish temperature. And, as will be further explained below, the austenite finish temperature does not always correlate with mechanical bending properties. Accordingly, a finding that “endodontic instruments have austenite finish temperatures above body temperature” is not sufficient to satisfy the claim limitation. Dr. Goldberg’s interpretation of the wherein clause is not reasonable.

47. I understand that Dr. Goldberg has considered the prosecution history of

the ’773 patent in forming his opinions. Dr. Goldberg relies on a document attached to an “Applicant Initiated Interview Request Form” that was submitted to the Patent Office on July 24, 2013. Ex. 1002, ¶¶ 33-34 (citing Ex. 1008 at 144-60).

48. I have reviewed Exhibit 1008, including the document attached to the Interview Request Form. Ex. 1008 at 144-60. In my opinion, nothing in that document makes Dr. Goldberg’s interpretation of the “wherein” clause reasonable. The wherein clause of the ’773 patent requires that the files heated according to the claimed method must have “an angle of greater than 10 degrees of permanent deformation” when tested in accordance the bending test described in ISO 3630. It is my understanding that Dr. Luebke was distinguishing his invention, which is a method for making a deformable nickel titanium endodontic file for use in performing root canal therapy, from the prior art reference, which disclosed a superelastic Nitinol wire, ribbon, sheet, or tubing. *Id*. at 104. As can be seen from the examiner’s summary of the interview with Dr. Luebke, the prior art conducted heat treatments for annealing and shape setting purposes to arrive at a superelastic device, whereas Dr. Luebke’s claimed invention pertained to heat treatments on a superelastic device (file) that resulted in a permanently deformable file. *Id*. at 163.

49. I understand that Petitioner also intends to rely on statements made during the prosecution history of a related patent, U.S. Patent No. 8,876,991 (the “’991 patent”), which it asserts support an interpretation that an A_f above body

temperature will satisfy the claimed permanent deformation requirement. I disagree. During prosecution of the patent application, Dr. Luebke submitted a declaration in which he stated:

> 5. The DSC of the endodontic instruments heat-treated at 375°C, 400°C, and 500°C all showed that the endodontic instruments were in the martensitic phase. These DSC results are attached as Exhibits A and B and C. This indicates that the endodontic instruments heat treated at 375°C, 400°C and 500ºC will all have an angle greater than 10 degrees of permanent deformation after torque at 45° of flexion when tested in accordance with ISO Standard 3630-1 as recited in pending independent claims 1, 6 and 11 of my patent application.

Ex. 1030 at 125-132. As an initial matter, where the A_f is above body temperature, it does not follow that the nickel titanium will be martensitic at room temperature. Depending on the other transition temperatures, such as A_s, M_s and M_f (and R_s and R_f), the nickel titanium can be biphasic. Biphasic nickel titanium can have superelastic properties. Ex. 2033 at 2:24-29. Therefore, I do not believe Dr. Luebke's statement provides any support for asserting that a file is permanently deformable, as recited in the claims, where the Af is above body temperature.

50. Additionally, without adequate testing to establish correlations for specific nickel titanium alloys, one cannot predict mechanical behavior based solely on crystal structure. Dr. Goldberg agreed to as much during his deposition in the district court litigation. Ex. 2025 at 87:3 - 88:4; 112:20 - 114:24; 123:21 -

124:14; 176:6-20. I understand Dr. Luebke has a great deal of experience testing his particular files. I also understand that he performed informal bend tests after conducting the heat treatments referred to in his declaration in Exhibit 1030. Ex. 2027 at ¶¶ 51-60. Based on his experience with prior tests (both DSC and bend tests), and his informal inspection and testing of the files, I believe he was reasonably able to predict the behavior of those files. But it is my opinion, consistent with Dr. Goldberg's original deposition testimony in the related litigation, that one cannot make an accurate prediction regarding mechanical behavior based on DSC testing alone without such additional testing.

The Kuhn reference does not anticipate the claims of the '773 patent

51. As an initial matter, I understand from reading Dr. Lemon's declaration, that in the period of over 15 years between the publication by Walia in 1988 (referenced above) and the filing of Dr. Luebke's international patent application in 2005, nobody thought to heat treat an entire superelastic nickel titanium endodontic file in order to make a softer, permanently deformable endodontic file. Ex. 2028, ¶ 33.

52. I have reviewed the Declaration of A. Jon Goldberg (Ex. 1002) that I understand to have been submitted by Petitioner US Endodontics, LLC in support of its petition for *inter partes* review. I understand that Dr. Goldberg offered an

opinion that Kuhn *et al*., 28 J. Endodontics 716 (2002) (Ex. 1019) ("Kuhn") discloses all of the elements in claims 1, 2 and 9-12 of the '773 patent. I disagree with Dr. Goldberg. In my opinion, Kuhn does not teach or suggest all of the elements of claims 1, 2, and 9-12 of the '773 patent. As described in detail below, Kuhn does not teach or even suggest heat treating an endodontic file to make it permanently deformable, and certainly does not teach or suggest: (1) heat treatment of the entire shaft of an instrument; or (2) a method of heat treatment that generates files having "an angle of greater than 10 degrees of permanent deformation" when subjected to bend testing in accordance with ISO 3630.

53. Kuhn states that the aim of their study was to show the fatigue characteristics of superelastic NiTi and the effect of the *process history* on the fracture life of endodontic files. Ex. 1019 at 716. In my opinion, one of ordinary skill in the art would understand that "process history" refers to the thermal cycling of cold working and/or annealing the nickel titanium alloy during the process of shaping an ingot into a wire, as well as changes that later occur in a file as a result of the repeated use in a clinical setting.

54. Kuhn states: "In the present work, fatigue properties of NiTi engine-driven rotary files have been characterized by using differential scanning calorimetry (DSC) and mechanical testing (bending)." Ex. 1019 at 716. "The DSC technique was used to measure precise transformation. The degree of deformation

by bending was studied with combined DSC and mechanical property measurements." *Id*. "DSC allows the identification of crystallographic phases at various temperatures." *Id*.

55. Kuhn studied instruments from Maillefer (ProFile) and Micro-Mega (Hero). *Id*. at 716-17. Some of the files tested by Kuhn were new, some of the files tested by Kuhn had been used in a clinical setting (described as 10-12 root canals followed by 5-6 autoclaves), and one of the files (the Profile 04./20) was subjected to a series of thermal heat treatments. The thermal treatments on the Profile 04/20 specimens "consisted of anneals at 350°C, 400°C, 450°C, 510°C, 600°C, and 700°C in salt baths for 10 min and at 600°C and 700°C for 15 min with the same process and subsequent water quench in all cases." *Id*.

56. Kuhn states that the "[s]pecimens were cut to separate the working or active part of the file from the inactive part." *Id*. at 717.

57. The transformation temperatures of the files in Kuhn were measured using DSC. *Id*. Kuhn cut 5 mm segments from each sample and obtained DSC thermograms. *Id*. Kuhn used the following DSC protocol: the cut specimens were placed in a DSC testing chamber (an aluminum pan) and then put in the DSC instrument; the DSC environment was warmed to 60°C and then cooled to -120°C at a cooling rate of 5°C/minute, and then the samples were heated to 60°C using the same rate. *Id*. Kuhn states that the start and finish temperatures of each phase

transformation were determined from tangent lines where the DSC curve deviates from the adjacent baselines. *Id*.

58. The DSC results are presented in Figures 1-4. The thermograms in Figure 1 compare the active parts of new Hero files of different conicity. The thermograms in Figure 2 compare the active and inactive parts of new ProFile 0.04/20 files. The thermograms in Figure 3 compare the active part of new and used Profile 0.06/20 files. And the thermograms in Figures 4A and 4B compare the active parts of a new ProFile 0.04/20 file with the active part of new ProFile 0.04/20 files subjected to heat treatment at 400°C, 510°C, 600°C, and 700°C in salt baths for 10 or 15 minutes.

59. To measure flexibility, Kuhn used a "bending testing machine" in which "the instruments were loaded with the same deformation." Ex. 1019 at 717. "The loading and the unloading were performed in the same conditions." *Id*. Kuhn states that they "obtained information about the elastic behavior (flexibility) of files and about heat treatments and clinical use." *Id*. Regarding the specifics of the bend testing, Kuhn explains only:

> At first, and until 3 mm of strain, only the tip of the instrument is bent. Then, between 3 and 6 mm, the curvature is in the middle of the file. Finally, above 6 mm, the part that has the maximum cross-sectional area near the handle becomes deformed in turn.
>
> As can be seen from the curves, <u>the samples</u> deformed at room

> temperature recover their original state, indicating that the transformation temperature is close to room temperature.

Id. at 718 (emphasis added).

60. In my opinion, one of ordinary skill in the art would understand that Kuhn did not perform the ISO 3630 bend test. As noted above, the ISO 3630 bend requires that the tip of a file be set 3 mm into a chuck on a torque-measuring device. The testing apparatus is then set to stop the angular deflection at 45°. A catch pin is mounted on the motor shaft of the device, and upon activation of the device, the catch pin will press against the shaft end of the instrument to bend the file until it reaches an angular deflection of 45°. By contrast, the bending testing machine in Kuhn appears to grasp the shank near where the handle was (i.e., before it was removed), and then bend the file from the tip end. Further, Kuhn states nothing about an angular deflection of 45° (or any other angle).

61. Kuhn conducted bending tests on the following file specimens: (a) ProFile 0.06/20 new; (b) ProFile 0.06/20 used; (c) ProFile 0.04/20; (d) ProFile 0.04/20 heat-treated at 400°C for 10 min.; (e) ProFile 0.04/20 heat-treated at 510°C for 10 min.; ProFile 0.04/20 heat-treated at 600°C for 15 min.; and ProFile 0.04/20 heat-treated at 700°C for 10 min. The results of the bending tests are presented in Figures 5, 6A, and 6B. The y-axis is force; *i.e.*, the load applied to the file. The x-axis is deflection, or the distance the file was deformed.

62. Kuhn explains that the results from the bending tests are qualitative: "The results are discussed only in a qualitative analysis and not a quantitative analysis because of the shape of the instruments (range and machining design), which prevents any calculation." Ex. 1019 at 717 (emphasis added). For example, Kuhn explains that the specimens show an increased flexibility for heat treatments below 600°C, and an increased stiffness for heat treatments above 600°C. Ex. 1019 at 719, 720 (Figure 6 legend). The qualitative analysis in Kuhn, therefore, is the relative effect of annealing conditions on the stress-strain behavior. *Id*. at 719.

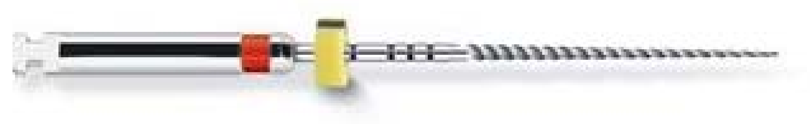
The "entire shank" limitation

63. In my opinion, one of ordinary skill in the art would conclude that Kuhn does not teach or suggest each element in the claims of the '773 patent because Kuhn did not perform step (b) in claim 1: "heat-treating *the entire shank*." Kuhn states that the "[s]pecimens were cut to separate the working or active part of the file from the inactive part." *Id*. at 717. In my opinion, the legend of Table 1 in Kuhn indicates that the *active* parts of the ProFile 0.04/20 files were subject to thermal treatments.[3] In my opinion, Kuhn does not heat treat the entire shank in

[3] ISO 3630, the international standard for root canal instruments, defines: "working part" as the portion of the root-canal instrument with an active cutting surface. Ex. 1017, 2-3; *see also* Ex. 1016, 2-3. The Profile .04/20 has a length of

any of his experiments.

64. ProFile instruments have been available since 1996. *See* https://www.dentsply.co.uk/Products/Endodontics/Endodontic-Files/Rotary-Files/ProFile.aspx (last visited Oct. 13, 2015). The image below depicts a ProFile with a handle affixed to a portion of the shank. *Id*.



65. Dr. Goldberg incorrectly asserts: "Because Kuhn is silent on the subject, one of ordinary skill in the art would have understood that the 'entire shank' was treated." Ex. 1019, ¶ 132. Dr. Goldberg also incorrectly asserts that the statement "near the handle" is an indication that Kuhn heat treated the entire shank. *Id*. (Citing Ex. 1019, 718). Dr. Goldberg further incorrectly asserts that special techniques would be needed to treat only a portion of a file. *Id*. At his deposition, Dr. Goldberg stated that the basis for his opinion that Kuhn thermally treated the entire files was the description of the bend test. Ex. 2034 at 61:2-8.

66. I disagree with Dr. Goldberg on all of these points. First, Kuhn is not "silent." Dr. Goldberg ignores Kuhn's explicit statement that the files were cut

---

25 mm. The "working part" or "active part" of the Profile .04/20 shank that contains the cutting surface is approximately 16 mm long.

with a diamond saw to separate the active from inactive part. Ex. 1019, 717. That statement is in the "Materials" section of Kuhn, which also teaches which files were tested (ProFile, Hero) and which tests were conducted on the each of the "specimens." *See* Ex. 1019 at Table 1 caption ("Table 1. NiTi specimens"). Dr. Goldberg also overlooks the statement in Table 1 that unequivocally indicates that only the active portion of the ProFile.04/20 was heat-treated. Table 1, as noted above, includes a legend, which uses a triangle symbol (▲) for "instruments after thermal treatments, *active part*." Ex. 1019, 717 (emphasis added). Kuhn Table 1 establishes that the ProFile 0.04/20 instruments were the only files studied after thermal treatments and only the "active part" of the ProFile 0.04/20 instruments was thermally treated. Therefore, Kuhn is not "silent" with respect to heat treating the *entire* shank. Rather, Kuhn explicitly states that the *active part* of the file was thermally treated.

TABLE 1. NiTi specimens

Conicity-Diameter	Hero	ProFile
0.02/30	◆	
0.04/20		◆ ✲ ▲
0.04/30	◆	◆
0.06/20		◆ ☆
0.06/30	◆	◆

◆ = new instrument, active part; ✲ = new instrument, inactive part; ☆ = used instrument, active part; ▲ = instrument after thermal treatments, active part.

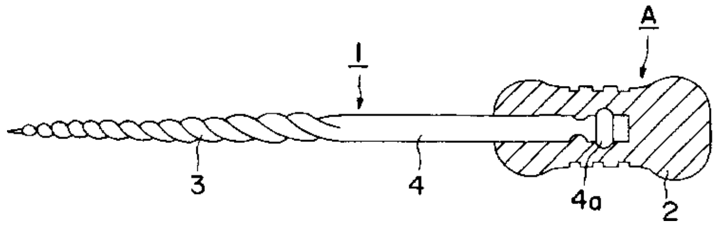
Ex. 1019 at 717.

67. Moreover, Dr. Goldberg is incorrect that Kuhn would require special

techniques to heat treat a portion of the file, because Kuhn has already separated the working portion before heat treatment. Once the shank had been cut to separate the working or active part of the file from the inactive part, as described by Kuhn, no other special technique would be required to heat only that portion of the shank.

68. In my opinion, one of ordinary skill in the art reading Kuhn would understand that Kuhn separated the "active part" of the ProFile.04/20 files from the remainder of the file and only heat-treated the active portion of the file, which he then subjected to DSC testing and bend testing. However, even if Kuhn were to interpret the terms "working part," "active part," or "inactive part" differently, such that the cut was made on the ProFile at the point where the handle intersects with the shank of the finished file, it is clear that at least a portion of the nickel titanium shank inside the handle was removed from the file prior to heat treatment.

69. It is my understanding that during the process of assembling an endodontic file, the shank is inserted into the handle of the instrument and secured in place, such that a portion of the shank remains recessed in the handle during use. The graphic below generally depicts an endodontic file having a portion of the shank inside the handle.



70. For a typical 25 mm file, the "active portion" of the file having cutting edges, would be approximately 16 mm in length. Ex. 1016, 4-5 (Fig. 1 (l_3) and Table 3 (l_3)). And as shown in the Figure above, a file has a substantial amount of the shank inside the handle of the instrument, and that portion of the shank would be removed along with the handle. Therefore, regardless of which way one interprets the terms "working part," "active part" and ""inactive part," it is clear that the thermal treatments in Kuhn were not conducted on the "entire shank" as required by claim 1 of the '773 patent.

71. Second, Dr. Goldberg's reliance on Kuhn's description of the bending tests—specifically, the statement "near the handle" on page 718—does not indicate that the handles were on the files. Kuhn expressly states that the files were cut before heat treatment. That statement is merely a reference to which end Kuhn appears to have clamped for the testing. Even if read differently, that passage suggests *at most* that the files may have been cut closer to the proximate end (*i.e.*, near the handle) than to the cutting edge part of the shank. Therefore, Dr. Goldberg's assumption that Kuhn did not remove the handle prior to bend testing is not supported by Kuhn.

The "10 degrees of permanent deformation" limitation

72. In my opinion, one of ordinary skill in the art would also conclude that Kuhn does not teach or suggest each element in the claims of the '773 patent because none of the files that were thermally treated above 400°C in Kuhn demonstrate permanent deformation after bending. Thus, Kuhn does not teach or suggest the following clause in the claims the '773 patent: " the heat-treated shank has an angle greater than 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO Standard 3630-1."

73. There are three portions of Kuhn that support my opinion. First are the DSC thermograms for the file specimens in Kuhn in Figure 4A and Figure 2. Second is the bending curve for the same file in Figure 6A. Third is Kuhn's explanation of the bending test results.

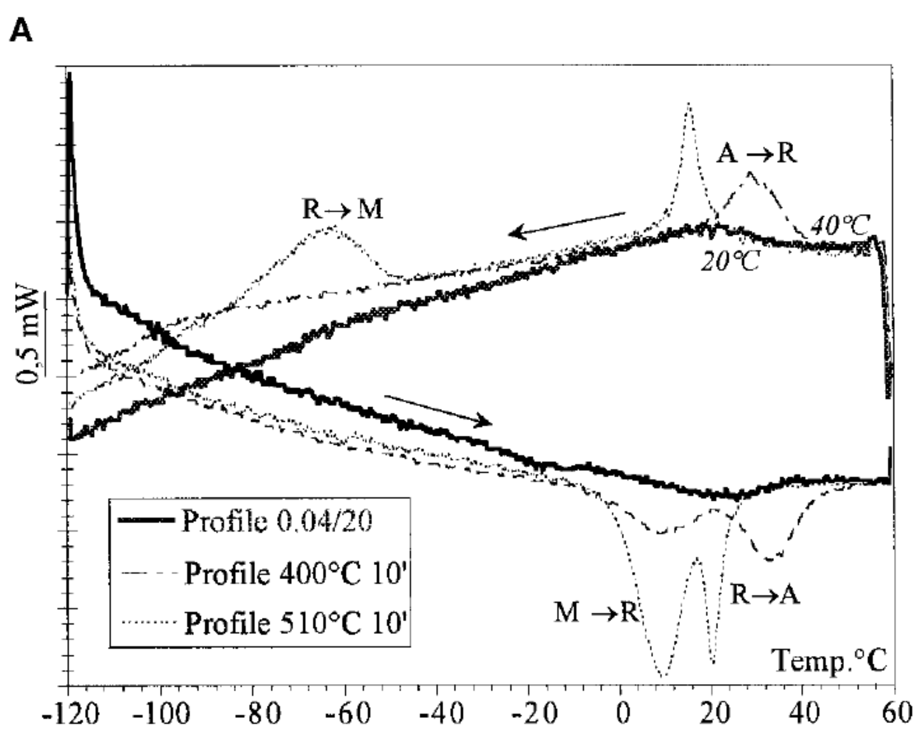
DSC thermograms

74. At the outset, one of ordinary skill in the art would not assume that the file was superelastic or permanently deformable based on a reported DSC thermogram (thermograms are informally referred to as "curves"). I disagree with Dr. Goldberg's statement that the "wherein" clause in the '773 patent claims can be satisfied if the prior art teaches heat-treated nickel titanium endodontic instruments that have austenite finish temperatures above body temperature. Ex. 1002 at 39. Rather, to determine the mechanical properties of an endodontic file or

whether the file would satisfy the "wherein" clause in the claims the '773 patent, one of ordinary skill in the art would conduct bend testing on the endodontic file. With that said, it is my opinion that one of ordinary skill in the art would glean the following information from DSC curves in Kuhn.

75. Kuhn Figure 4A (shown below) shows DSC thermograms (heating and cooling directions) for: (1) the active portion of the file heat-treated at 400°C for 10 minutes; (2) for the active portion of the file heat-treated at 510°C for 10 minutes; and (3) untreated, superelastic ProFile 0.04/20.

Kuhn Figure 4A



76. The heating curve for the heat-treated file specimens are described as a transition from martensite (M) to R-phase (R) and from R to austenite (A).

Ex. 1019, Fig. 6A. That transition is shown to occur between 0-40°C for both files. The cooling curve for the ProFile 0.04/20 heat-treated at 400°C for 10 minutes shows a transformation between 15-40°C. The cooling curve for the ProFile 0.04/20 heat-treated at 510°C for 10 minutes shows a transformation between 5-25°C, and another transformation between -50 and -90°C.

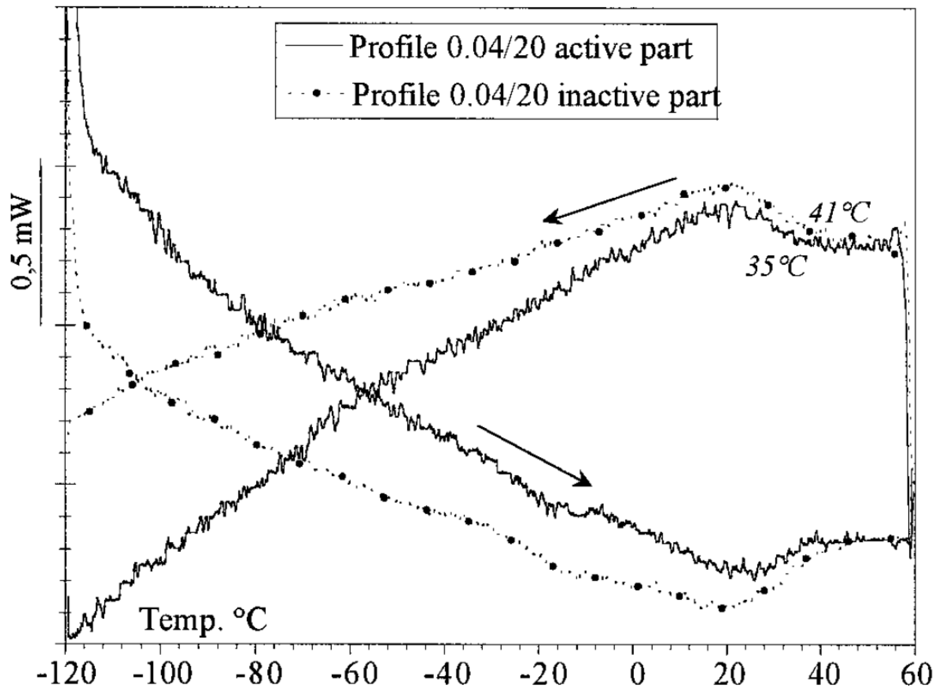
77. The DSC heating curves for each heat treated file suggest that a martensitic transformation occurs close to room temperature. The heating curve in Kuhn Figure 4A for the file specimen that was heat treated at 400°C suggests that the file specimen is likely to be biphasic at and around room temperature, which means that it has some austenite and either R-phase or martensite present.

78. In my opinion, one of ordinary skill in the art would not be able to determine whether the heat treated files in Kuhn would meet the "wherein" clause in the claims the '773 patent relying only on the DSC thermograms because the materials appear to be biphasic at or around room temperature. It was known in the art that nickel titanium can exhibit superelastic behavior well below its A_f temperature, such as when it is biphasic. Ex. 2033 at 2:24-29.

79. Indeed, the DSC curves in Figure 2 suggest that the untreated file specimen is *also* biphasic at and around room temperature, having some austenite and either R-phase or martensite present. Kuhn Figure 2 shows heating and cooling curves for the active part of an untreated (*i.e.*, no heat treatment) ProFile

0.04/20. The DSC heating curve shows a martensitic transformation occurring between 0-40°C. Kuhn Figure 2 is reproduced below.

Kuhn Figure 2



80. In my opinion, one of ordinary skill in the art would not rely only on the DSC thermograms in Kuhn to establish that the files meet the "wherein" clause in the claims the '773 patent.

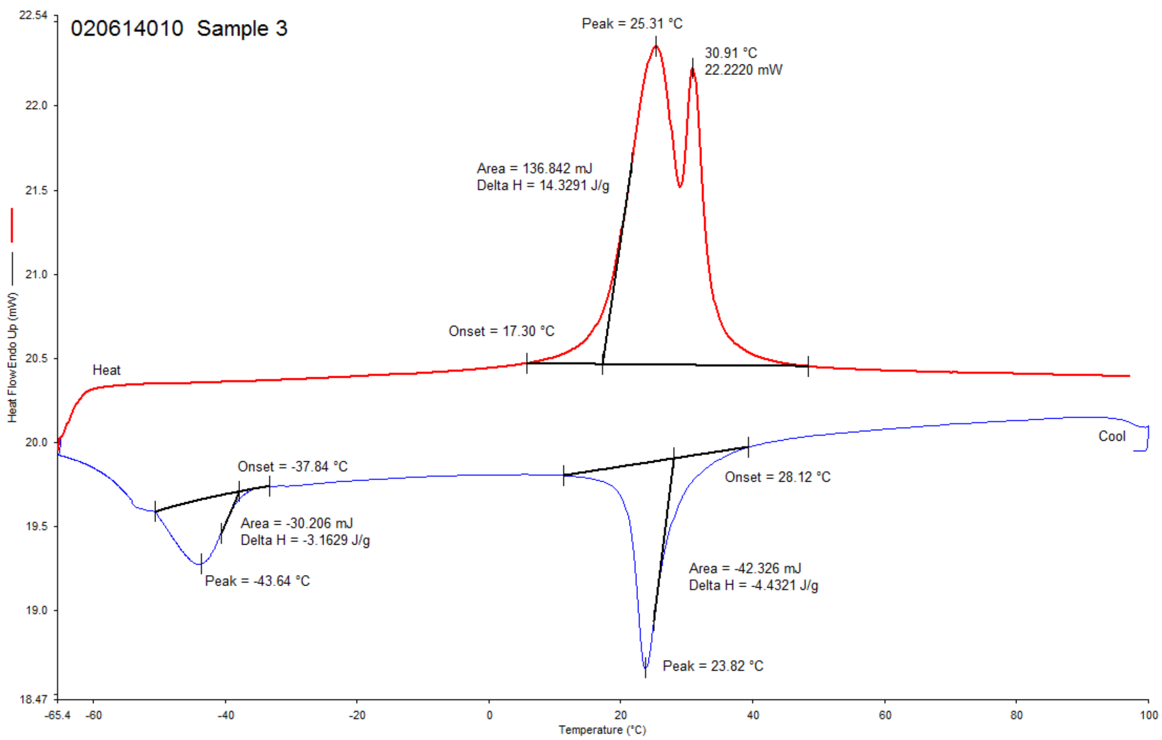
81. It should be noted that Kuhn does not *prove* the crystal structure of the tested endodontic files. Kuhn only provides DSC data, which is used to *describe* the transformation between crystal structures in the tested materials. One of ordinary skill in the art would not accept DSC data as conclusive proof of a crystal structure. The Kuhn authors, however, published an earlier paper which provides

additional insight as to the crystal structure of the tested endodontic files. Kuhn *et al.*, *Influence of Structure on Nickel-Titanium Endodontic Instruments Failure,* J. Endod. 2001, 27, 516-20 ("Kuhn 2001") (Ex. 2024). Kuhn 2001 discloses x-ray diffraction (XRD) experiments on the files. Ex. 2024 at 517. The thermal treatments in Kuhn 2001 are identical to the thermal treatments in Kuhn. Ex. 2034 at 69:3-22. Kuhn 2001 explicitly states that the files are austenite. *Id*. ("Results of the XRD of Hero and Profile files showed that the alloys are fully austenite at room temperature."). Table 1, which includes the XRD data, also states that the tested files are austenite. *Id*. This is supported by the SEM testing in Kuhn 2001, where the files had to be clamped to maintain a bend (suggesting the files were superelastic). One of ordinary skill in the art would understand that the combined Kuhn and Kuhn 2001 disclosures refute Dr. Goldberg's assertion that the file heat treated at 400°C would meet the "wherein" clause of the claims the '773 patent. That is, regardless that the 400°C heat treatment increased the A_f of the ProFile from 35°C to 40°C, one of ordinary skill in the art would evaluate the totality of experimental evidence and conclude that that file would not meet the "wherein" clause of the claims the '773 patent.

82. Additionally, the Kuhn DSC thermograms for the file specimen heat treated at 400°C are similar to DSC thermograms from other nickel titanium endodontic files that I reviewed. Exhibit 2030 (Ex. A) is a DSC testing report

conducted on five different files sold by Edge Endo, one of the real parties in interest for Petitioner.

83. The DSC thermograms for the EdgeFile X3 file specimens also suggest a martensitic transformation at or around room temperature. The EdgeFile X3 files were heat treated at 500°C. Ex. 2001 (Second Substitute) at 257:1-6. Shown below are DSC thermograms for an EdgeFile X3 file specimen (Sample 3).



84. The DSC data is displayed such that the heating curve on top (in contrast to Kuhn, which displays the heating curve on the bottom). The curves suggest that at room temperature the files were biphasic, having some austenite and either R-phase or martensite present.

85. In my opinion, one of ordinary skill in the art would recognize the

similarities between DSC thermograms obtained from the EdgeFile X3 file specimen and the DSC thermogram obtained for the file specimen heat treated at 400°C in Kuhn. For example, the DSC heating curve thermograms include a two-step martensitic transformation at or around room temperature. As will be discussed further below, the EdgeFile X3 files were subjected to the bend test required in claim 1 of the ’773 patent and did not maintain at least 10 degrees of permanent deformation after deflection at 45 degrees. Accordingly, as noted above, one of ordinary skill in the art would not assume that the file was superelastic or permanently deformable. Rather, in my opinion, one of ordinary skill in the art would need to conduct bend testing on the file to determine its mechanical properties.

Bending test results

86. As noted above, one of ordinary skill in the art would understand that the bending tests in Kuhn are not the same as the bending test in ISO 3630. The Kuhn bending curves describe a stress-strain test, where a specimen from the files is subjected to a load to bend it, and then the load is withdrawn.

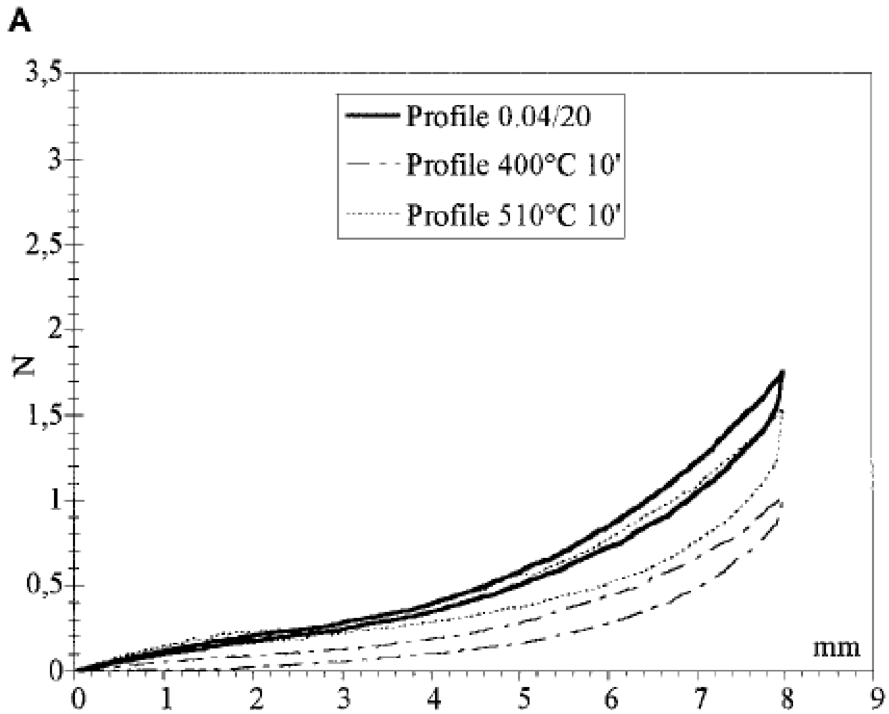
87. Each of the bending curves in Kuhn Figures 5, 6A, and 6B shows a reversible transformation consistent with superelastic files. Specifically, the curves in Figures 5, 6A, and 6B plot force against deflection. Ex. 1019 at 719-20. The file specimens were deflected 8 mm during loading, and upon unloading they

recovered their original shape and revert back to the y-axis origin. *Id*.

88. Petitioner and Dr. Goldberg have conceded that the file specimen heat treated at 510°C recovers its original shape. Pet. at 32; Ex. 1002, ¶ 140. However, Dr. Goldberg asserts that the file specimen heat treated at 400°C does not recover its original shape, and would meet the "wherein" clause limitation in the claims of the '773 patent.

89. Dr. Goldberg is wrong. Kuhn Figure 6A is shown below. In my opinion, one of ordinary skill in the art would understand Kuhn Figure 6A to mean what it shows: the bending curves return to the y-axis. A return to the y-axis indicates that the files recover their original (straight) shape after the load is removed. In my opinion, one of ordinary skill in the art would understand Kuhn Figure 6A to mean that the heat treated files remained superelastic after heat treatment.

Kuhn Figure 6A



90. None of the curves in Figures 5, 6A, and 6B intersect the x-axis. Therefore, one of skill in the art would understand that none of the file specimens in Kuhn, including those heat-treated at 400°C or 510°C, would demonstrate an angle greater than 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO 3630.

91. Dr. Goldberg mischaracterizes the curve in Kuhn Figure 6A. He then relies on that mischaracterization to allege that the file specimen heat treated at 400°C would meet the permanent deformation limitation in claim 1 of the ’773 patent. Specifically, Dr. Goldberg alleges that the bending curve “intersects” the x-axis at 1.8 mm. Dr. Goldberg then uses the 1.8 mm number, which is an

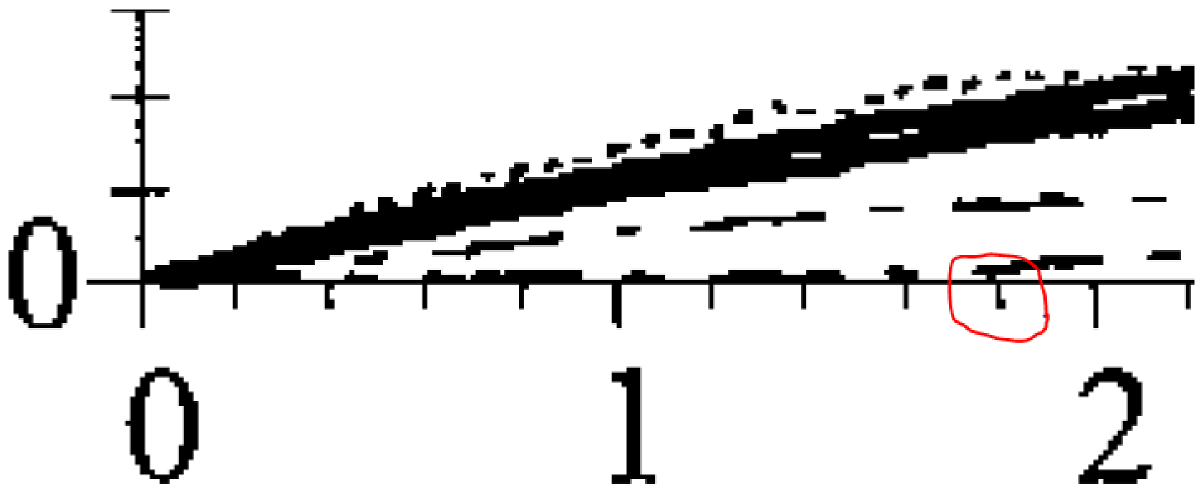
incorrect interpretation of Fig 6A, as a basis for what he refers to as a "rough estimate" of what one could expect if Kuhn had used the ISO 3630 bend test. Ex. 1002, ¶ 135.

92. In my opinion, Dr. Goldberg's analysis of Figure 6A is wrong. He interprets the bending curve of the file specimen heat-treated at 400°C as "intersect[ing] the x-axis at a point other than the origin." Ex. 1002, ¶ 135. But the curve never intersects the x-axis. And the only thing close to the x-axis near 1.8 mm is a stray pixel. Dr. Goldberg's attempt to correlate his interpretation of Kuhn Figure 6A to the ISO 3630 bend test is based on one pixel.

93. As just noted, Dr. Goldberg incorrectly interprets Kuhn Figure 6A to "intersect" the x-axis at 1.8 mm. Besides one stray pixel,[4] circled in red below, the curve for the ProFile 0.04/20 heat-treated at 400°C for 10 minutes does not coincide with the x-axis at 1.8 mm. Rather, as can be seen in the enlarged figure above, the curve sits on top of the x-axis and returns to the point of origin.

---

[4] Other stray pixels can be seen, among other places, on the y-axis itself and on the markings for the x-axis (at 0.2, 0.8, 1.8).



94. In my opinion, one of ordinary skill in the art would conclude that the curve for the file specimen heat-treated at 400°C to *intersect* the x-axis. In fact, it returns to point of origin just like those of the other files. Since the curve returns to the y-axis, one of ordinary skill in the art would conclude that the file recovers its original shape.

95. Further, it would not be possible for one of ordinary skill in the art to correlate the data from the Kuhn bend test to what would be expected if these same files were tested using the ISO bend test based on the little information provided in Kuhn—especially when one does not know for certain how the bend test in Kuhn was conducted.

96. My opinion of Kuhn Figure 6A is also informed by (1) how others in the art report bending curves; and (2) the combined DSC and bending test results of the EdgeFile X3.

97. The bending curves in Kuhn Figure 6A are consistent with bending curves reported for other superelastic nickel titanium materials. For example, in Miura (Ex. 1004), the authors reported bending curves for orthodontic wires that had been heat treated at 400°C. Miura states that the 400°C heat treatment had no significant effect on the mechanical properties of the wire. Ex. 1004 at 4. The bending curves are shown in Figure 5 (reproduced below).

Miura Figure 5

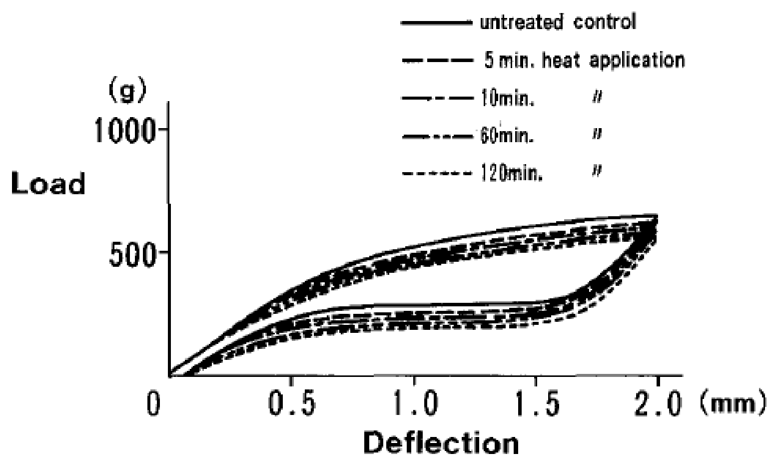


Fig. 5. Results of the heat application on 0.016 inch Japanese NiTi alloy wire at 400° C.

Ex. 1004, 5. Each curves in Figure 5 returns to the y-axis or nearly to the y-axis (consistent with permanent set) upon unloading. Accordingly, the heat treatment in Miura at 400°C had no significant effect.

98. The bending curves in Kuhn Figure 6A are <u>in</u>consistent with bending curves reported for nickel titanium materials that are not superelastic. When non-superelastic nickel titanium alloys are deformed by a bending test, the curve will

intersect the x-axis. For example, Miura Figure 3 shows that superelastic nickel titanium alloys return to the y-axis during unloading, whereas stainless steel and Co-Cr-Ni alloys intersect the x-axis.

Ex. 1004 Figure 3

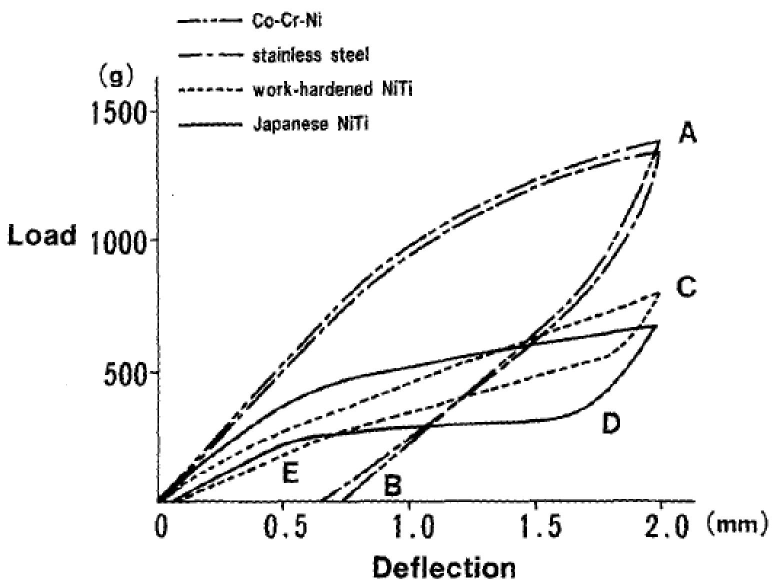
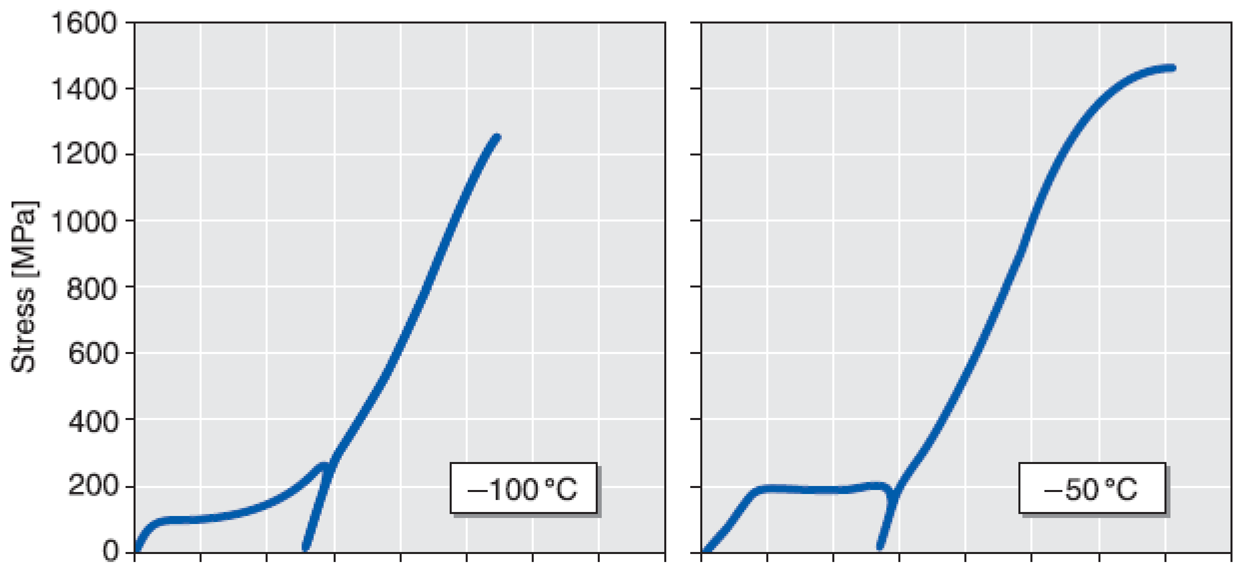


Fig. 3. Load deflection curves produced by the three-point bending test.

Ex. 1004 at 4.

99. As another example, Pelton Figure 5 shows that wires tested at -100°C and -50°C are not superelastic and intersect the x-axis. "The tensile curves shown in Figure 5 illustrate that the mechanical behavior of Nitinol varies greatly from 100°C to 150°C. In these tests, wires with an As of -22°C and Af of 11°C were pulled to 6% strain, unloaded to zero stress and were then pulled to failure. At the lowest test temperatures, the wires are martensitic and the high residual strains are fully recovered by heating above Af (the shape-memory effect)." Ex. 1006 at 111.

Pelton Figure 5 (in part)



Ex. 1006 at 111.

100. In view of Kuhn, Miura, and Pelton, one of ordinary skill in the art would understand that when a bending curve returns to the y-axis upon unloading, it indicates that the alloy recovers it original shape. Thus, it is my opinion that one of ordinary skill in the art would interpret the bending curves in Kuhn Figure 6A, including that for the 400°C anneal, to represent a superelastic nickel titanium material that recovers its original shape.

101. As noted above, my opinion of Kuhn Figure 6A is also informed by the DSC and bending test results of the EdgeFile X3. The DSC results are discussed above. The EdgeFile X3 files were subjected to the ISO 3630 bend test in claim 1 of the ’773 patent. Exhibit 2030 (Ex. A) is a testing report prepared by Knight Mechanical Testing.

102. As noted in the report, the testing was conducted in accordance with ISO 3630. Exhibit 2030 (Ex. A). The EdgeFile X3 files did not maintain at least 10 degrees of permanent deformation after deflection at 45 degrees. *Id*. Three samples were tested, and each of the EdgeFile X3 files recovered their original shape (albeit with *de minimus* bend). The angle of permanent deformation was measured to be about 1-4 degrees. Shown below is an image of EdgeFile X3 (Sample 3) with its handle removed, after the ISO 3630 bend test.

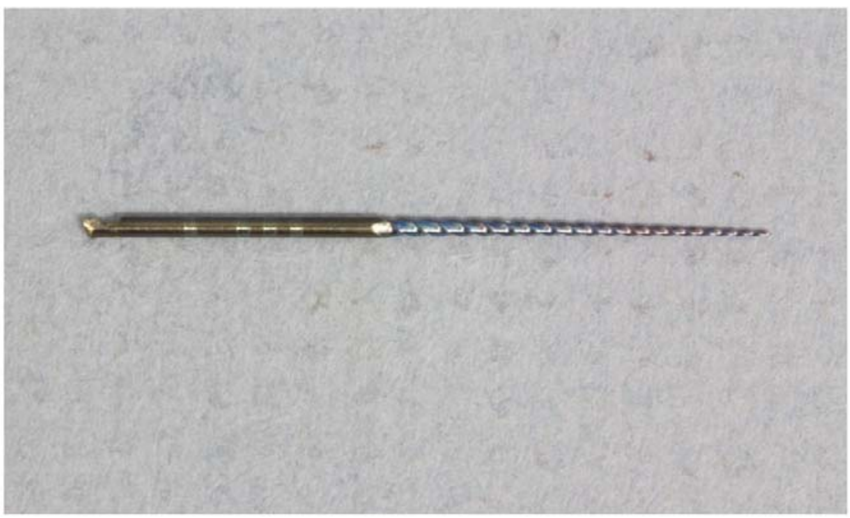


Figure 7: Posttest sample X3_S3

103. Other EdgeFiles were also tested. Those EdgeFiles were also heat treated at 500°C. Ex. 2001 (Second Substitute) at 257:1-6. But in contrast to the EdgeFile X3, the EdgeFile X1 files were permanently deformed after the ISO 3630 bend test. The image below shows an EdgeFile X1 (sample 1) with its handle removed, after the ISO 3630 bend test. The angle of deformation for the EdgeFile X1 files was determined to be about 23-27 degrees.

Figure 2: Posttest sample X1_S1

104. In my opinion, one of ordinary skill in the art would have concluded that the file specimens heat treated in Kuhn are superelastic and would not meet the "wherein" clause in the claims of the '773 patent.

105. Kuhn refers to the heat treatments as anneals (Ex. 1019 at 717), and recommends "thermal treatments at approximately 400°C (recovery) before machining to decrease the work hardening of the alloy . . ." (Ex. 1019 at 720). Annealing and recovery anneals are terms of art that are (and were as of 2002) understood to refer to heat treatments to impart or maintain superelasticity while removing dislocations. U.S. Patent No. 5,843,244 demonstrates this general understanding. Ex. 2033 at 2:5-13 (referring to the annealing step as the "so-called 'recovery' processes."). This is consistent with Kuhn's usage of the term, and a person of skill in the art would have understood the thermal treatments in Kuhn to be recovery anneals that generally would be understood to not affect the nickel

titanium's superelasticity significantly. For this additional reason, I do not believe that Kuhn made a permanently deformable endodontic file as recited in the claims of the '773 patent. Instead, Kuhn suggests heat treatments to do the opposite, *i.e.* maintain superelasticity.

Kuhn's explanation of the bending test results

106. Finally, my opinion that the file specimens heat treated in Kuhn are superelastic and would not meet the "wherein" clause in the claims of the '773 patent is informed by a statement in Kuhn that requires little interpretation. Kuhn expressly states that "the samples deformed at room temperature recover their original state, indicating that the transformation temperature is close to room temperature." Ex. 1019, 718. Kuhn's statement is not accompanied with exceptions or notes suggesting that the file specimen heat-treated at 400°C is an outlier. Accordingly, there is no basis to conclude that the files were deformed.

107. Kuhn's statement is consistent with the DSC thermograms. As discussed above, the heating curve for the file specimen heat-treated at 400°C shows a transformation at or around room temperature. Kuhn's statement is also consistent with the bending curve in Figure 6A, which shows a curve for the file specimen heat-treated at 400°C returning to the y-axis. That curve indicates that the file specimen recovered its original shape.

Dr. Goldberg's Interpretation of Kuhn Is Wrong

108. Dr. Goldberg's convoluted testimony regarding Kuhn is confusing, inconsistent with his prior statements, and contrary to how one of ordinary skill in the art would understand Kuhn. For the following reasons, one of ordinary skill in the art would not agree with Dr. Goldberg's opinion that Kuhn anticipates the invention claimed in claim 1 of the '773 patent.

109. *First*, Dr. Goldberg applies an unusual definition of superelasticity to suggest that the DSC data reported for the file specimen heat-treated at 400°C means that the file specimen is not superelastic and therefore would meet the permanent deformation limitation in claim 1 of the '773 patent.

110. Specifically, Dr. Goldberg incorrectly asserts that Kuhn defines superelastic alloys as having a transition temperature range (TTR) lower than mouth temperature. Ex. 1002, ¶ 131 (citing Ex. 1019 at 716). Kuhn's discussion of superelasticity is provided below:

> The superelasticity (SE) nature of NiTi has been attributed to a reversible austenite to martensite transformation. It is believed austenite is transformed to martensite during loading and reverts back to austenite when unloaded. The transformation is reversible during clinical use, because SE alloys have a transition temperature range (TTR) lower than mouth temperature.

Ex. 1019 at 716. Contrary to Dr. Goldberg's assertion, this is not a definition of superelasticity. It is a comment regarding the transition temperature range of superelastic alloys.

111. Dr. Goldberg's reading of Kuhn is not only erroneous but also contrary to the normal definition of superelasticity, which Dr. Goldberg recited pretty accurately in paragraph 27 of his declaration (shown below), except, as noted above "original shape" includes a small amount of permanent set.

> Superelasticity means that the material is relatively rigid until a threshold stress is applied to it; above that threshold, the material because considerably more flexible. When the stress is removed, the material reverts to its original shape.

Ex. 1002, ¶ 27; Pet. at 3.

112. Dr. Goldberg also incorrectly asserts that one can determine whether a material is superelastic or deformable based only on the material's austenite finish temperature. Ex. 1002, 39; Ex. 2034 at 31:2-7. As discussed above, that is not correct. Moreover, Dr. Goldberg repeatedly conceded at his deposition in the related district court litigation that one of ordinary skill in the art *cannot* determine the mechanical properties of a nickel titanium alloy using DSC alone. Ex. 2025 at 87:3-88:4; 112:20 - 114:24; 123-124; 176. Rather, he conceded that one would need to conduct the bend testing. *Id*. Indeed, Dr. Goldberg himself defines superelasticity to mean that the material reverts to its original shape when bent. Ex. 1002, ¶ 27.

113. One of ordinary skill in the art would not conclude that a material is superelastic based only on thermal testing with DSC. One of ordinary skill in the art would also test the mechanical properties of the material to determine whether it is superelastic. Specifically, one would apply a load to the material and determine if it recovers its original shape after the load is removed.

114. Furthermore, Dr. Goldberg's definition of superelasticity (in his declaration) is unworkable. Ex. 1002, ¶ 131. Dr. Goldberg testified on cross examination that it would be impossible for a nickel titanium alloy to demonstrate superelastic properties if it were tested at a temperature between its austenite start temperature and its austenite finish temperature. Ex. 2034 at 16:2 - 17:11. But after Dr. Goldberg was directed to Pelton Figure 5, which shows nickel titanium wire that was tested at 0°C and 10°C has superelastic properties below its austenite finish temperature (22°C), and the new, untreated ProFile in Kuhn, which he said had an A_f around 36°C yet had superelastic properties at room temperature, he conceded that his analysis was flawed. Ex. 2034 at 55:8 - 58:6.

115. *Second*, Dr. Goldberg's refers to a "transition temperature" throughout his declaration in reference to Kuhn without ever clarifying *which* transition temperature he is referring to. I understand that he did not clarify his opinions at his deposition. Ex. 2034 at 37:6 - 50:15. When asked to explain which transition temperature he was referring to in several of those paragraphs, Dr. Goldberg could

not do so. *Id*. In fact, Dr. Goldberg admitted that he did not understand what Kuhn was referring with respect to "transition temperature." Ex. 2034 at 54:11-13.

116. Dr. Goldberg acknowledges that there are at least four temperatures used to describe the transformation between the austenite and martensite crystalline phases in nickel titanium: martensite start, martensite finish, austenite start, and austenite finish. Ex. 1002, ¶¶ 28-29. And Dr. Goldberg's interpretation of the "wherein" clause in claim 1 of the '773 patent requires a determination of one of those four temperatures: *the austenite finish temperature*. Ex. 1002, ¶ 39.

117. Dr. Goldberg testified that he did not determine the austenite finish temperature for any of the DSC thermograms in Kuhn *prior* to his deposition. Ex. Ex. 2034 53:17-22. Instead, Dr. Goldberg relied on the "transition temperatures" reported in Kuhn Table 3. Dr. Goldberg bases his opinions of Kuhn on the "transition temperature." For example, see Ex. 1002 at ¶¶ 131, 137, 152, 155, 157, 160, 166, 185.

118. *Third*, a person of ordinary skill in the art could not correlate the results from the Kuhn bend test to another, such as the ISO 3630 bend test, because there is not sufficient information provide by Kuhn that would allow one to make such a correlation.

119. Dr. Goldberg's "rough estimate" (Ex. 1002, ¶ 135) is not sound for a number of reasons: (a) Dr. Goldberg does not know how the bend test was

performed in Kuhn; (b) Dr. Goldberg has not established that the geometries used in the Kuhn bending test are the same as the geometries used in the ISO Standard 3630-1 bending test (45 degree bend); (c) Dr. Goldberg's correlation relies on the incorrect assumption that the curve "intersects the x-axis at about 1.8 mm"; and (d) Kuhn draws the curve back to the y-axis. Rather, the only thing that one skilled in the art can glean from Kuhn Figure 6A is that the file specimen heat-treated at 400°C is more flexible than both the file specimen heat-treated at 510°C and the unheated file.

120. Further, Dr. Goldberg asserts that the bending test in Kuhn would produce greater permanent deformation than the ISO 3630 bending test because ISO 3630 test holds the tip end of a file and the Kuhn test holds the shank end of a file. Dr. Goldberg incorrectly alleges that holding the tip end would create a fulcrum that increases the stress on the nearby material. Ex. 1002, ¶ 136. It is not possible to make a statement to this effect absent knowing how Kuhn performed the bend test. This is simply conjecture.

121. Additionally, Kuhn reported a test of one file. One cannot draw any conclusions based on one file. Figure 6 of the '773 patent illustrates the need for a proper sample size. The figure shows that TT files maintained about 28-30 degrees of permanent deformation after testing in accordance with ISO 3630. Ex. 1001, Figure 6. The figure includes an error bar denoting about 2 degrees of variance for

those files. The Ti-N coated files maintained about 15-24 degrees of permanent deformation after testing in accordance with ISO 3630. The figure includes an error bar denoting about 2-10 degrees of variance for those files. In my opinion, one of ordinary skill in the art would not, and could not, draw the conclusions that Dr. Goldberg has from only the one sample in Kuhn.

122. *Fourth*, Dr. Goldberg incorrectly asserts Kuhn's statement that "the samples deformed at room temperature recover their original state," applies only to the curve for the untreated, superelastic file in Figures 5, 6A, and 6B—but not the other curves for the heat-treated ProFiles in Figures 6A and 6B. Ex. 1002, ¶ 131. Dr. Goldberg provides no basis or explanation for his interpretation. In my opinion, each of the curves in Figure 6 is consistent with a superelastic file that recovers its original shape, and I interpreted this statement to be referring to all of the files, including the file specimens that were heat treated.

123. *Fifth*, Dr. Goldberg did not consider the Kuhn's counterpart article, Kuhn 2001. Ex. 2024. As discussed above, the authors performed the same heat treatments on the same endodontic files and concluded based on, *inter alia*, x-ray diffraction (XRD) experiments, that the ProFiles were austenite. Ex. 2024, __. This is supported by the fact that the files had to be clamped for SEM testing, suggesting the files were superelastic.

The claims of the ’773 patent are not obvious in view of Kuhn and ISO 3630-1

124. I disagree with Dr. Goldberg that the invention claimed in claims 8, 13, 15, and 17 of the ’773 patent would have been obvious in view of Kuhn and ISO 3630 (Ex. 1016).

125. As discussed above, one of ordinary skill in the art would understand that Kuhn does not teach or suggest all of the elements in the claimed invention. In particular, Kuhn does not teach or suggest thermal treatments on the entire shank of an endodontic file, and Kuhn does not teach or suggest thermally treated root canal instruments that maintain at least 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO 3630. On the contrary, after discussing the thermal treatments in the study, Kuhn concludes: “Thus, the stiffness is much more important. For clinical applications, these heat treatments are not required.” Ex. 1019 at 719-20. In my opinion, one of ordinary skill in the art would understand Kuhn to suggest that stiffer endodontic files are preferable, and that heat treatments are not required to obtain that desired feature.

126. In the concluding paragraph, Kuhn also provides some suggestions to improve the lifetime of endodontic files, including applying thermal treatments at approximately 400°C (recovery) <u>before machining</u> to decrease the work-hardening of the alloy . . .” *Id*. at 720. Therefore, in my opinion, one of ordinary skill in the art would understand that Kuhn expressly teaches away from the process claimed

in the ’773 patent, which requires heat treatment after machining, and deformable files. Further, the “recovery” anneals that Kuhn is discussing were generally understood to be heat treatments to remove dislocations without significantly affecting the alloys superelasticity. Ex. 2033 at 2:5-17.

127. I disagree with Dr. Goldberg that Kuhn’s explicit suggestion to thermally treat the alloy before machining does not “subtract” from what Kuhn actually did. Ex. 1002, ¶148 (citing Ex. 1019 at 720). In my opinion, one of ordinary skill in the art would read that passage from Kuhn in the context of the entire publication. As noted above, Kuhn states that “heat treatments are not required.” Ex. 1019 at 719. Moreover, Kuhn states: “Machining process promotes a high density of defects; the alloy is work-hardened.” *Id*. at 717. Kuhn, in the “Discussion” section, states again that machining “promotes work hardening and creates surface defects,” and that the active part of the file is more affected by machining than the inactive part. *Id*. at 719. Dr. Goldberg’s opinion neglects the fact that Kuhn conducted thermal treatments on machined files in order to address the issues created by machining. After conducting testing that included heat treatment and analyzing those results, Kuhn concludes, based on his investigation, that heat treatments are not necessary for clinical applications and suggests thermal treatments (recovery anneals understood to be designed so as to *not* affect superelasticity) to alloys <u>before machining</u>.

128. The combination of Kuhn with ISO 3630 does not teach the invention claimed in claims 8, 13, 15, and 17 of the ’773 patent. Section 4 of ISO 3630 is “Requirements,” and subsection 4.1 is “Material.” ISO 3630 requires that the instruments be made of stainless or carbon steel: “The working part and the shaft, if one part, shall be made of stainless steel or carbon steel.” Ex. 1016, 8 (§4.1.1).

129. Section 6.4 of ISO 3630 is entitled Resistance to bending. Ex. 1016 at 13. The testing apparatus and procedure are described in subsections 6.4.1 and 6.4.2, respectively. *Id*. The procedure requires removal of the handle from an instrument prior to testing, and once the tip of the instrument is secured, the opposite end is bent 45 degrees.

130. In my opinion, one of ordinary skill in the art would make the following observations: (a) ISO 3630 does not mention nickel titanium instruments; (b) ISO 3630 does not mention thermal treatments to endodontic files; (c) ISO 3630 does not teach or suggest a preference for any instrument or test; and (d) ISO 3630 does not teach or suggest an endodontic instrument that can maintain permanent deformation after testing with the procedure in subsection 6.4.2.

131. I disagree with Dr. Goldberg that claims 8, 13, and 15-17 of the ’773 patent are obvious. Ex. 1002, ¶¶ 139-144. Dr. Goldberg does not identify any teaching from Kuhn or ISO 3630 that suggests the claimed invention. In my opinion, claims 8, 13, 15 and 17 would not have been obvious in view of Kuhn and

ISO 3630. Further, Dr. Goldberg's premise that Kuhn teaches the desirability of a file with an A_f is incorrect. The transition temperatures reported in Table 3, upon which Dr. Goldberg relies (*e.g.*, Ex. 1002 at ¶ 137), are not A_f temperatures. They correspond to transition temperatures on the cooling curve. Thus, Dr. Goldberg's statement that an A_f is desirable based on Kuhn is wrong.

The claims of the '773 patent are not obvious in view of Kuhn, ISO 3630, McSpadden, and Pelton

132. I disagree with Dr. Goldberg that the invention claimed in claims 1-17 of the '773 patent would have been obvious in view of Kuhn, ISO 3630, US Patent Application Publication No. US 2002/0137008 (Ex. 1022) ("McSpadden"), and Pelton *et al.*, *Min. Invas. Ther. & Applied Technol.* 2000, 9(1), 107-118 ("Pelton"). In my opinion, claims 1-17 would not have been obvious in view of Kuhn, ISO 3630, McSpadden and Pelton.

133. McSpadden is a published patent application directed to endodontic files having a "relatively high loading plateau greater than about 500 MPa." Ex. 1022, Abstract, ¶ 15; *see also* claims 1-14 on p. 7 (claiming an endodontic instrument with a loading plateau greater than about 500 MPa, 750 MPa, and 850 MPa in claims 1-3, respectively). McSpadden describes certain problems observed with nickel titanium files in the background section of the application. Specifically, McSpadden states that nickel titanium instruments have become "widely accepted

in the industry," but come with certain drawbacks. *Id*. at ¶ 10. McSpadden states that it is difficult and expensive to machine nickel titanium endodontic files. *Id*. at ¶¶ 11, 42. During the machining process, deposits and/or imperfections along the cutting edges form, which affect the performance of the files. *Id*. at ¶¶ 12, 43. Other drawbacks are that nickel titanium files have reduced sharpness compared to stainless steel files. *Id*. "Another ***significant drawback*** is reduced manipulation control due to reduced stiffness and extreme torsional flexibility of presently available NiTi endodontic files as compared with stainless steel files." *Id*. (emphasis added).

134. After describing the problems in the art, McSpadden describes his invention as "an improved class of <u>superelastic</u> alloys and manufacturing techniques . . . which overcome the aforenoted drawbacks while <u>preserving and enhancing the essential advantages of the superelastic alloy</u>." *Id*. at ¶ 44 (emphasis added). McSpadden states "that by increasing the loading plateau of a superelastic alloy, its machinability, cutting-edge sharpness and sharpness holding-ability, and manipulation control are improved." *Id*.

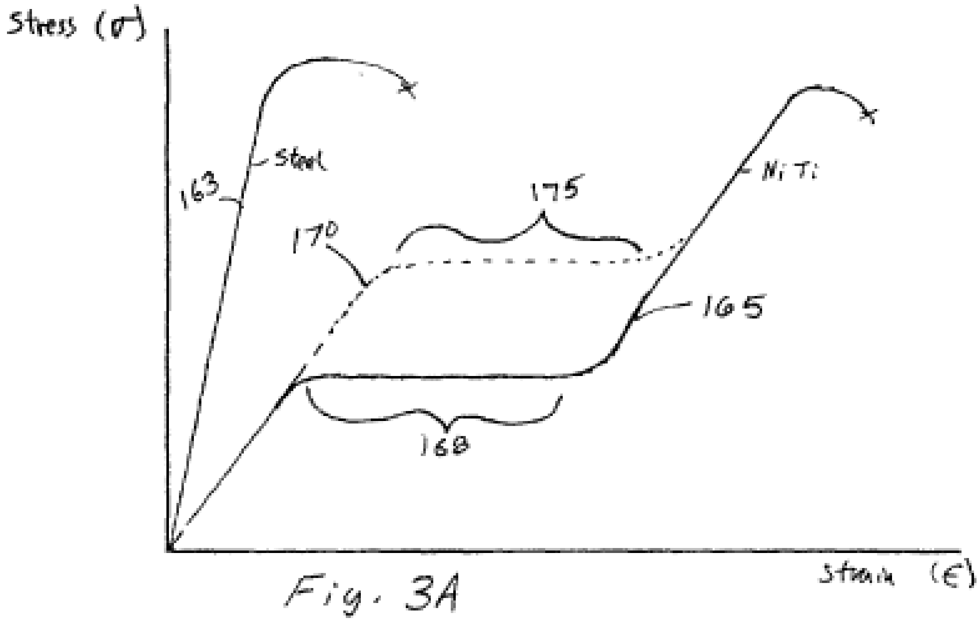
135. McSpadden explains that in conventional manufacturing processes, the nickel titanium alloy wire is heat treated and annealed "prior to machining." *Id* at ¶ 51. McSpadden found that a "certain amount of residual cold working" (also performed prior to machining) can improve the machining characteristics. *Id*. ¶ 52.

136. The only experiments that McSpadden conducted with respect to heat treating endodontic files after machining are described as "selective heat treatment and/or annealing," which is "accomplished by passing an electrical current through a length of cold-work NiTi wire" or through an endodontic file. *Id*. at ¶¶ 59, 63. McSpadden thus does not teach or suggest any of the temperature limitations in any of the claims.

137. In my opinion, a person of ordinary skill in the art would understand McSpadden as providing a method of manufacturing nickel titanium alloys that are easier to machine and superelastic endodontic instruments that are stiffer. To accomplish those goals, McSpadden cold-works the alloy prior to machining. McSpadden finds that superelastic, stiffer (less flexible) files improve responsiveness and control of the file during clinical use.

138. McSpadden Figure 3A is reproduced below. Figure 3A is a comparative stress-strain diagram of various nickel-titanium alloys versus stainless steel. Ex. 1022, ¶ 23.

McSpadden Figure 3A



139. McSpadden discusses Figure 3A in ¶¶ 46-48. Based on the following passages, one of ordinary skill in the art would understand the invention discussed in McSpadden to be a superelastic nickel titanium endodontic file having greater stiffness than the superelastic nickel titanium endodontic file that were available at the time.

140. *First*, McSpadden explains what the loading plateau is: "As can be discerned from [Fig. 3A], each of the NiTi alloys has a significantly larger strain to failure than stainless steel. This is largely because the NiTi alloys exhibit a superelastic property that enable them to undergo significant strain at a substantially constant stress or load level, sometimes called the 'loading plateau' **168**, **175**." Ex. 1022, ¶ 46.

141. *Next*, McSpadden states that his invention has a higher loading plateau than other superelastic nickel titanium endodontic files: "Curve **165** indicates a stress-strain curve and loading plateau **168** for a typical NiTi alloy, such as SE508, used to fabricate endodontic files. Curve **170** indicates a stress-strain curve and loading plateau **175** for an improved class of superelastic alloys having features and advantages in accordance with the present invention. In particular, those skilled in the art will recognize that the curve **170** indicates a higher loading plateau **175** than the loading plateau **168** of curve **165**." *Id*. at ¶ 47.

142. *Finally*, McSpadden explains why a superelastic nickel titanium endodontic file having a higher loading plateau is desirable: "The increased loading plateau **175** is desirable for several reasons. For a given file design and diameter, a higher loading plateau increases the apparent stiffness of the file (in both bending and torsion) and therefore its responsiveness and ease of manipulation by endodontists. Files formed from conventional NiTi alloys can often feel overly flexible and non-responsive and, thus, exhibit reduced tactile feedback and difficult manipulation control—particularly in the smaller diameter files. Endodontic files fabricated from improved superelastic alloys having increased stiffness in accordance with one preferred embodiment of the invention provide improved responsiveness and manipulation control without significantly adversely increasing the risk of file breakage or canal wall transportation." *Id.* ¶ 48.

143. In my opinion, one of ordinary skill in the art would understand that McSpadden is describing a superelastic nickel titanium endodontic file having greater stiffness. At the same time, one of ordinary skill in the art would understand that McSpadden is teaching that "overly flexible" files are non-responsive and exhibit reduced tactile feedback and difficult manipulation control. McSpadden criticizes conventional NiTi files as being "overly flexible," "non-responsive" and difficult to manipulate. McSpadden's criticism of an overly flexible file exemplifies the conventional wisdom that Dr. Luebke fought against when trying to market his invention—that decreasing superelasticity and increasing flexibility is not better.

144. While McSpadden Figure 3A does not include units on either axis, it clearly displays a superelastic stress-strain curve (having a loading and unloading plateau).[5] Moreover, McSpadden Figure 3A and claims 1-14 (claiming an endodontic instrument with a loading plateau greater than about 500 MPa, 750 MPa, and 850 MPa in claims 1-3, respectively) are consistent with Pelton Figure 5, which does display units for the stress (MPa). Ex. 1006 at 111-12.

145. Pelton Figure 5 shows that the loading plateau increases with testing temperature. Specifically, the loading plateau is about 500 MPa for the alloy tested at 0°C, about 600 MPa for the alloy tested at 40°C, and about 700 MPa for the

[5] Compare McSpadden Fig. 3 with, *e.g.*, Pelton Fig. 4 (Ex. 1006 at 111).

alloy tested at 60°C. To obtain the curves in Figure 5, the alloy wire was pulled to a 6% strain, unloaded to zero stress and were then pulled to failure. Ex. 1006 at 111.

Pelton Figure 5

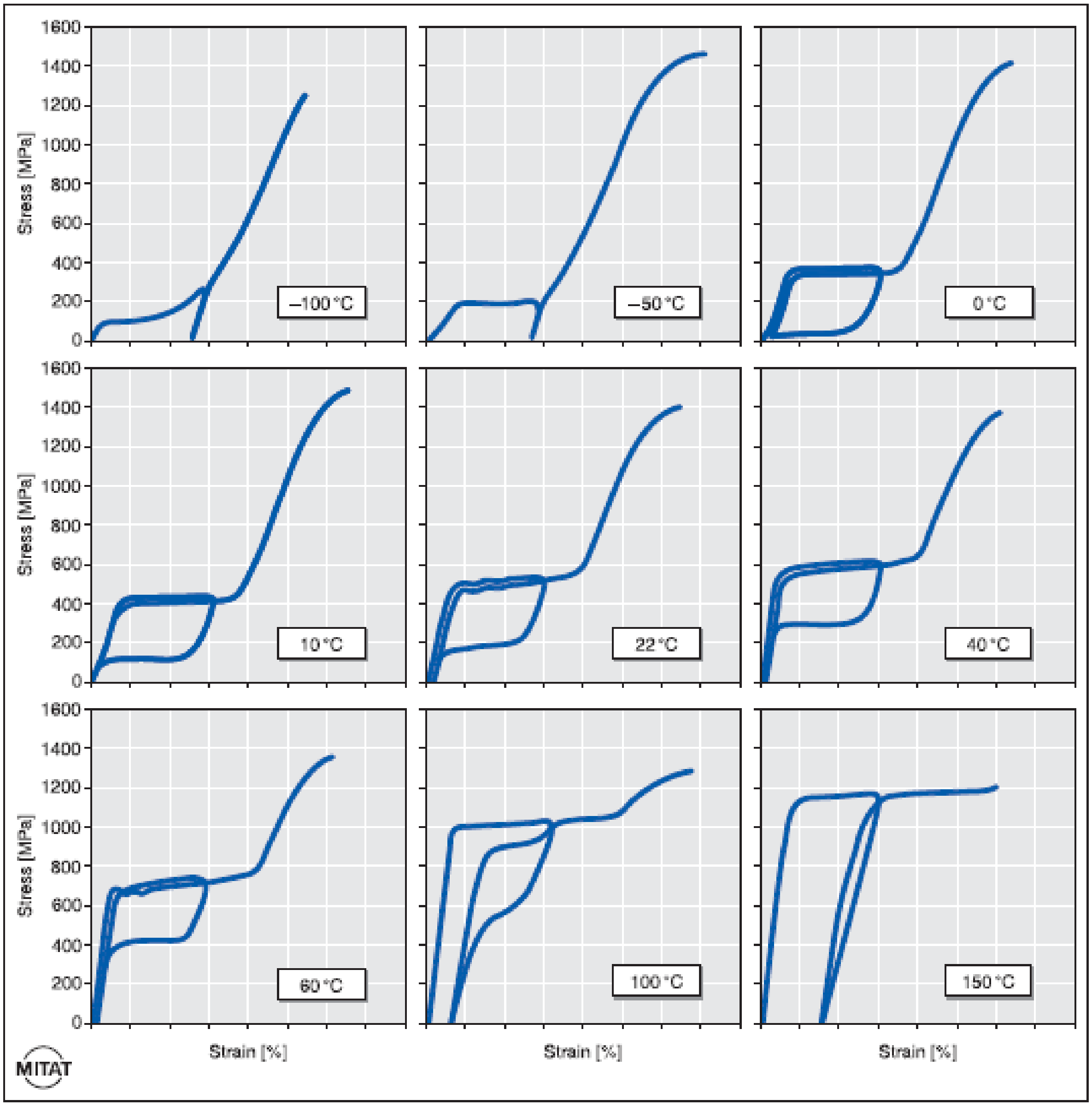


Figure 5. Effect of test temperature on the mechanical behaviour of Nitinol wire. Note that there is a systematic increase in the loading and unloading plateau stresses with increasing test temperature. Below 0°C, the structure is martensite and, above 150°C, the graph shows conventional deformation of the austenite. The intermediate temperatures all show classic transformational superelasticity.

146. McSpadden also states than when a file is heat-treated after machining, a heat treatment via electric current is used. Since electric current is used, McSpadden does not mention or suggest a temperature for heat treatment. Specifically, McSpadden states that "heat treatment and/or annealing is selectively applied or otherwise conducted in a manner that significantly anneals and releases latent crystalline deformation within the central or core area **465** but does not significantly anneal and release latent crystalline deformation within the surrounding jacket of material **470** immediately adjacent the outer surface thereof." *Id*. at ¶ 63. In my opinion, one of ordinary skill in the art would understand that McSpadden is disclosing stiffer (less flexible), superelastic endodontic instruments. One would not read McSpadden as suggesting the invention claimed in the '773 patent, which heat treats the entire shank of a superelastic file to obtain a file that maintains at least 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO 3630.

147. Dr. Goldberg's opinion relies exclusively on the statement in ¶ 52 without regard to how the problem in the art was addressed by McSpadden or what McSpadden was trying to accomplish (cold working an alloy to achieve a stiffer file after machining). Specifically, Dr. Goldberg relies on the following statement: "If desired, the formed endodontic file (*i.e.*, subsequent to machining) may be further heat treated and/or annealed in order to achieve the desired degree of

superelasticity or other material properties and/or to set a desired file shape (straight, pre-curved or pre-twisted).” *Id*. at ¶ 52.

148. Although McSpadden mentions post-machining heat treatment in passing, he states that it is either “to achieve the desired degree of superelasticity” or “to set a desired shape.” *Id*. Regarding the former, I explained above that any post-machining heat treatments are thought to be selective to the core area and do not treat the surrounding area, or the entire shank. McSpadden teaches that the purpose of this type of heat treatment is to obtain a stiffer file. McSpadden does not mention or suggest a temperature for heat treatment. Regarding the latter, a shape-set file, still being superelastic, would not meet the limitation requiring at least 10 degrees of permanent deformation in the ISO 3630 bend test. Shape setting is not permanent deformation. And McSpadden does not teach or suggest any particular temperature for the shape-setting step.

149. McSpadden is referring to U.S. Patent No. 5,843,244 (the “’244 patent”) to Pelton (Ex. 2033), which is incorporated by reference into McSpadden. Ex. 1022, ¶ 53. The ’244 patent explains that nickel titanium alloys are processed using cold working and annealing steps. Ex. 2033 at 2:5-17. The cold working step involves, for example, “swaging, drawing, pressing, stretching or bending.” *Id*. at 2:5-9. Following the cold working step is an annealing step, “at a temperature less than the recrystallization temperature of the alloy, for a time

sufficient to cause dislocations to rearrange, combine and align themselves (so-called 'recovery' processes)." *Id*. at 2:9-13. The '244 patent explains that "a device is to be [often] used in a shape other than that which can be practically produced by cold working." *Id*. at 2:34-36. Accordingly, "it is customary to form the drawn, cold worked wire into the desired 'heat stable' shape, to constrain the wire in that shape, and then to perform the above described recovery heat treatment to 'shape set' the component." *Id*. at 2:38-42. The '244 patent then states: "the final annealing operation has two purposes: to adjust the superelastic properties of the alloy, and to properly set the shape of the article." *Id*. at 2:42-44.

150. Dr. Goldberg's reliance on the statement in ¶ 52 of McSpadden is incomplete without a discussion of the '244 patent, which explains what McSpadden is suggesting in that paragraph. The '244 patent does not teach or suggest the manufacture of an endodontic file, or any other application, having the ability to be flexible and permanently deformable. Therefore, I disagree that a person of ordinary skill in the art would be motivated to obtain a file that maintains at least 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO 3630 based on the disclosure of McSpadden.

151. I disagree with Dr. Goldberg that one of ordinary skill in the art would have been motivated to combine McSpadden ¶ 52 with the teachings of Kuhn and Pelton. Ex. 1002, ¶ 149. In my opinion, one of skill in the art would not be

motivated to combine the teaching of McSpadden and Pelton with Kuhn.

152. As discussed above, Kuhn states: "Machining process promotes a high density of defects; the alloy is work-hardened." *Id*. at 717. Kuhn, in the "Discussion" section, states again that machining "promotes work hardening and creates surface defects," and that the active part of the file is more affected by machining than the inactive part. *Id*. at 719. Kuhn concludes that "heat treatments are not required." *Id*. So, Kuhn and McSpadden both discuss defects with respect to machined files. McSpadden teaches that the alloy should be heat-treated before machining and Kuhn teaches that heat treatments are not required after machining. Specifically, Kuhn suggests "thermal treatments at approximately 400°C (recovery) before machining to decrease the work-hardening of the alloy." *Id*. In view of the '244 patent, which McSpadden incorporates by reference, it becomes clear that Kuhn's use of "recovery" refers to introducing superelastic properties into nickel titanium. *See* Ex. 2033 at 2:5-17 ("A preferred way in which non-linear superelastic properties can be introduced in a shape memory alloy involves . . . The cold working step is followed by an annealing step . . . (sol-called 'recovery' processes)." Accordingly, I disagree that a combination of Kuhn and the passing discussion in ¶ 52 of McSpadden would have motivated a person of ordinary skill in the art to arrive at the claimed invention because those references essentially teach away from doing so. McSpadden's invention is nearly the *opposite* of the

claimed invention.

153. Additionally, Dr. Goldberg's supposed "motivation" for combining these references is the "good results" disclosed by Kuhn with a "transformation temperature" of 40°C. Ex. 1002, ¶ 150. As noted above, Kuhn did not teach or suggest an A_f of 40°C after a 400°C anneal. Thus, one of skill in the art would not have been motivated by Kuhn to make an endodontic file with an A_f of 40°C.

154. Pelton also does not provide any motivation for combining these references. Pelton is not directed to endodontic files. Rather, Pelton is a review article that discusses the austenite finish temperatures of nickel titanium alloy wire of a specific composition that was heat-treated at six different temperature points, spanning from 350°C to 550°C, at six different time points. Ex. 1006 at 114 (Figure 9). Pelton does not study heat treatments on finished products. He reviews studies on nickel titanium *wire* to understand how time and temperature affect its superelasticity. Ex. 1006 at 109 ("The focus will be on products that are superelastic between room temperature and body temperature."). Pelton expressly states that his paper's purpose is to review how nickel titanium *wire* is processed, and the properties of processed wire. *Id*. at 107. Further, Pelton seeks to understand how to *optimize*, not *lessen*, the superelastic properties of nickel titanium products. *Id*. at 107 (title: "Optimisation…"); *id*. at 108 ("Optimisation of the superelastic properties of Nitinol..."). The nickel titanium alloy products

discussed do not include endodontic files. *Id*. at 107.

155. In my opinion, Pelton does not meet a single limitation of claim 1. Pelton does not relate to endodontic instruments and thus does not teach step (a) or (b) of claims 1 and 13 of the '773 patent. Pelton does not analyze or discuss superelasticity or permanent deformation, and thus does not teach the "greater than 10 degrees of permanent deformation after testing in the ISO Standard 3630-1 bend test" limitation of those claims. One skilled in the art would not be motivated by Pelton to make a file a permanently deformable file, let alone a file that exhibits at least 10 degrees of permanent in the ISO 3630 bend test.

156. Pelton studies processing of nickel titanium <u>wire</u>. Applications for the wire mentioned in Pelton include endoscopic instruments, stents, filters, and orthodontic wire. Pelton does not discuss using the wire for endodontic files and does not contemplate or consider what effect the subsequent machining or grinding (to create the cutting edges on an endodontic file) could have on the material.

157. One of ordinary skill in the art would know that machining processes can affect the transformation temperature of nickel titanium alloys. Ex. 2032 at 1 (section 6.2); Ex. 2036 at 1293. Furthermore, as Kuhn demonstrates, there can be variability in the transformation temperatures for the "active portion" or working portion of the shank (where the cutting edges are located) and the transformation temperature in the "inactive portion" or non-working portion of the an endodontic

file. Ex. 1019 at 718 ("DSC cooling and heating curves in Fig. 2 show . .. two different TTR for the active part (35°C) and inactive part (41°C)"). Therefore, one of ordinary skill in the art would not necessarily expect the austenite finish temperature for a completed, machined endodontic file to be the same as the austenite finish temperature for a wire.

158. Furthermore, Pelton may be useful for what it teaches regarding optimization of superelasticity during processing of nickel titanium wire, but Pelton does not teach or suggest that permanent deformation is desirable in a finished endodontic file.

159. Kuhn also notes that "processing history," which would include machining, promotes work hardening and affects the files' properties, and the active part and inactive parts are affected differently. Kuhn at 717, 719.

160. Moreover, as explained above, one of ordinary skill would not rely solely on DSC testing generally, much less on a single transformation temperature in order to determine whether a finished file would exhibit the level of permanent deformation required by the claims of the '773 patent. Because it is possible for the material to be "bi-phasic" at certain temperatures, one would perform mechanical testing to determine whether a finished file would exhibit the claimed permanent deformation.

161. Dr. Goldberg relies on Pelton for the particular combinations of time and

temperatures at which Pelton performs his heating process. However, Pelton does not express any preference for heat treatment at any particular time or temperature. Nor does Pelton suggest that time and temperatures could be adjusted to achieve permanent deformation in a finished, machined, endodontic file. Thus, Pelton does not teach or suggest the invention of the ’773 patent—a process for heat treating the entire shank of a finished (machined) superelastic endodontic file in order to reduce its superelasticity and generate a file that is permanently deformable.

162. In my opinion, one of ordinary skill in the art would not be motivated to apply the heat treatments discussed in Pelton to endodontic files; *i.e.*, machined instruments. Dr. Goldberg has not cited to any portion of Pelton that suggests post-machining heat treatments. Indeed, Pelton provides no basis for applying the heat treatments discussed in Pelton to a machined file. For instance, Pelton does not specifically suggest 1-2 hours at 475-525°C or 1-2 hours at 500°C as heat-treatment times and temperatures, and thus would not motivate a person skilled in the art to choose those times and temperatures to achieve permanent deformation. Therefore, I disagree that a person of ordinary skill in the art would be motivated to obtain a file that maintains at least 10 degrees of permanent deformation after torque at 45 degrees of flexion when tested in accordance with ISO 3630 based on the combined teachings of Kuhn, ISO 3630, McSpadden, and Pelton.

The claims of the ’773 patent are not obvious in view of Matsutani, Pelton, and ISO 3630

163. I disagree with Dr. Goldberg that the invention claimed in claims 1-17 of the ’773 patent would have been obvious in view of Kuhn, ISO 3630, US Patent No. 7,137,815 (Ex. 1023) (“Matsutani”), and Pelton.

164. Matsutani is directed to the manufacture of root canal instruments. Ex. 1023 at 1:7-9. Matsutani defines the object of the invention as providing an endodontic file with segmented superelastic properties. Specifically, Matsutani states the object of the invention as “weakening the action of returning to an original shape of the tip portion of the work portion” and “the superelastic characteristic in the remaining portion.” *Id*. at 2:11-34.

165. To achieve the object of the invention, Matsutani teaches the following methods of selective heat treatment: (A) blowing cool air on one part of the file shank and hot air on another part; (B) insulating one part of the file shank and heating another part; (C) cooling one part of the file and heating another part; (D) using a cooling agent on one part of the file and heating another part; and (E) blowing cool air on one part of the file and heating another part. *Id*. at 6:54 - 7:19.

166. Matsutani does not provide temperatures for any of the disclosed methods.

167. Matsutani selectively heat-treats only the tip of a file and intentionally prevents heating the remainder of the working portion and shank, because heat-

treating any more than three-quarters of the working portion would make the file "difficult to cut the root canal well." Ex. 1023, 5:37-42. Specifically, Matsutani teaches that "if the shape memory portion 6 is larger than ¾ of the work portion, at the time of inserting the tip 3 into the root canal and rotating it, a problem may occur in that the position of a rotational axis is not fixed, but is made eccentric to make it difficult to cut the root canal well." *Id*. This is consistent with concerns expressed in other prior art references. *E.g.*, Ex. 1025 at 211 ("When flexible files were used in an attempt to traverse such canals, many would buckle, just like a wet noodle . . .."); Ex. 1022 at ¶12 ("Another significant drawback is reduced manipulation control due to reduced stiffness and extreme torsional flexibility of the presently available NiTi endodontic files as compared with stainless steel files.").

168. Dr. Goldberg admits that Matsutani only heat-treats part of the shank. Ex. 1002, ¶ 201. Dr. Goldberg also admits that Matsutani teaches away from heat-treating the entire shank. *Id*. at ¶ 202.

169. Dr. Goldberg's remaining discussion of Matsutani suggests that one of ordinary skill in the art would selectively take Matsutani's teaching about selectively heat treating the tip of a file but disregard Matsutani's rationale for doing so. Specifically, Dr. Goldberg discusses taper and rigidity and then suggests that basic engineering would have led a person or ordinary skill in the art to heat

treat the entire file, rather than just the tip as taught by Matsutani.

170. I disagree with Dr. Goldberg because he fails to account for the clinical application of the files. Dr. Goldberg ignores the problems in the art that Matsutani was addressing. Matsutani is concerned with root canal procedures. Matsutani explains that one of the problems with conventional root canal instruments is that when the tip portion is inserted into the root canal for the treatment of the root canal and is bent, "a repulsive force is generated to act on the wall of the root canal. Thus, there is an undesirable possibility that . . . the work portion might cut the outside of a bent portion of the root canal more heavily, and might cut the inside in the center of the bent portion more heavily, so as to penetrate the root canal." Ex. 1023 at 1:51-64. Matsutani also states that since the file is rotated in a state where the work portion is bent, "a repeated bending stress is applied to the work portion. This increases the possibility of breaking a slender tip portion of the work portion." *Id* at 1:65 - 2:7. Dr. Goldberg's discussion of taper and rigidity misses the point; he does not explain how it is relevant to the problem addressed by Matsutani.

171. I disagree with Dr. Goldberg that a later-filed Japanese patent application supports his theory: that because endodontic files are tapered and rigid, one of ordinary skill in the art would ignore the teachings in Matsutani for selective, partial heat-treatment of the tip of a file. Ex. 1002, ¶ 204. The later filed

application, Ex. 1027, does not support Dr. Goldberg's theory because in that application, Matsutani does *not* heat-treat the entire shank of the file *either*. Dr. Goldberg admits this. Ex. 1002, ¶ 205. Instead, Matsutani states that the "work portion 4" of a file may be heat treated. Ex. 1027 (throughout). The work portion is the machined portion of the file. *Id*. at 9 (Figure 1). Accordingly, the second Matsutani patent application does not suggest to heat treat the entire shank, as required by the process claimed in claims 1-17 of the '773 patent.

172. Dr. Goldberg incorrectly implies that one of skill in the art would not agree with Matsutani—which explains that heat treating the entire shank would create performance issues. His analysis is not sound for at least these reasons: (a) he asks one of ordinary skill in the art to discredit and ignore an explicit teaching in Matsutani; (b) he asserts that one of ordinary skill in the art would have a "reasonable expectation of success" for heat-treating the entire shank, however, he points to no evidence for that expectation in Matsutani, Pelton, or ISO 3630; (c) he does not identify any motivation in Matsutani, Pelton, or ISO 3630 for heat-treating the entire shank; (d) he relies on a later Matsutani reference that does not heat treat the entire shank; and (e) he ignores the clinical problem that Matsutani was trying to solve and instead incorrectly asserts that "it would have been simpler and cheaper to heat-treat the entire shank." Ex. 1002, ¶ 206.

173. Dr. Goldberg's taper analysis and reworking of the meaning of "working

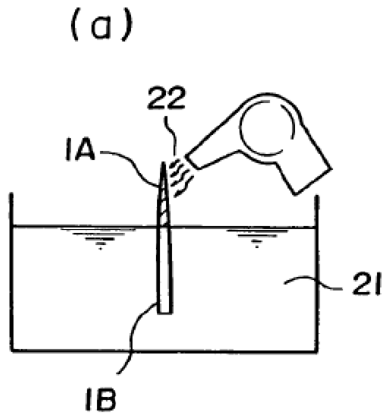
portion" (the portion of the file labeled "4" in Matsutani Figure 4) does not explain why one of ordinary skill in the art would disregard Matsutani's preference for partial heat treatment. Ex. 1002, ¶ 206.

174. As just noted, Dr. Goldberg incorrectly stated that one of ordinary skill in the art would take a divergent path from Matsutani because "it would have been simpler and cheaper to heat-treat the entire shank." However, I understand that Dr. Goldberg testified that he did not know the common ways of heat treating nickel titanium alloys. Ex. 2034, 84:4 - 85:13. I disagree with Dr. Goldberg's rationale because it has no basis. Matsutani does not state that the preferred heating methods are expensive or cumbersome. In fact, one of ordinary skill in the art would recognize that the methods discussed in Matsutani are cheap and easy.

175. As noted above, Matsutani teaches the following methods of selective heat treatment: (A) blowing cool air on one part of the file shank and hot air on another part; (B) insulating one part of the file shank and heating another part; (C) cooling one part of the file and heating another part; (D) using a cooling agent on one part of the file and heating another part; and (E) blowing cool air on one part of the file and heating another part. *Id*. at 6:54 - 7:19.

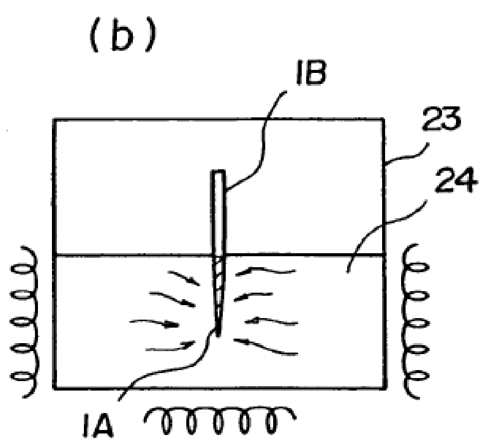
176. Matsutani Figure 4 provides an illustration of those methods.

Matsutani Figure 4a



"A portion **1B** not to be heat-treated is partially dipped in water **21** as a cooling agent so as to prevent a temperature increase or is blown by cool air and, at the same time, a portion **1A** to be heat-treated is blown by hot air **22**, thereby being partially heated." Ex. 1023 at 6:54-60.

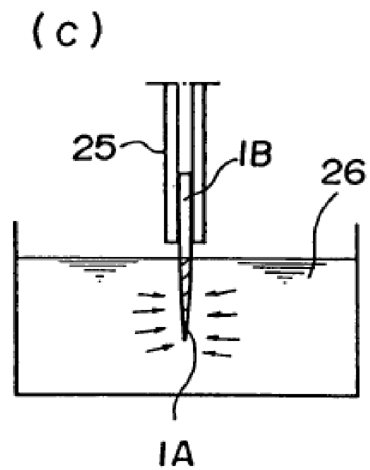
Matsutani Figure 4b



"A furnace **23** is partitioned into two layers and a portion **1A** to be heat-treated is partitioned by heat insulating material **24** so as to prevent a portion **1B** not to be heat treated from being increased in temperature and is heated in the furnace **23**.

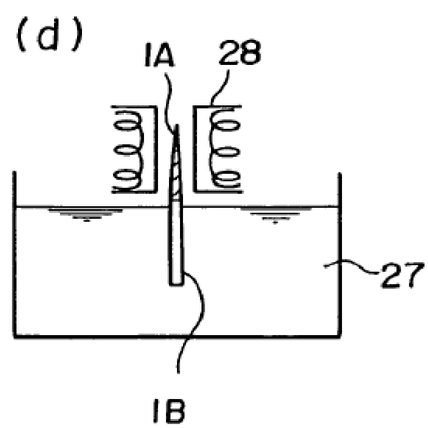
Here, if a portion **1A** can be surely insulated from heat, the furnace **23** is not required to be partitioned." Ex. 1023 at 6:54-60.

Matsutani Figure 4c



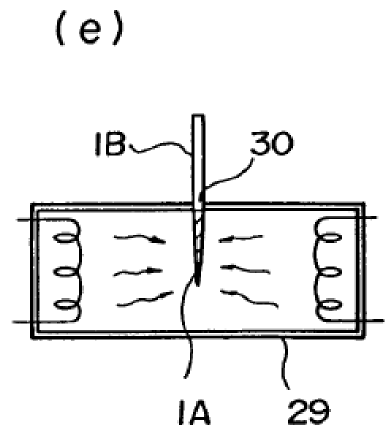
"A portion **1B** not to be heat-treated is held by a clip **25** provided with a cooling function, and a portion **1A** to be heat-treated is dipped in liquid in a salt bath **26** at high temperatures, thereby being heated." Ex. 1023 at 7:1-5.

Matsutani Figure 4d



"A portion **1B** not to be heat-treated is dipped in a cooling agent **27**, such as water, to prevent a temperature increase, and a portion **1A** to be heat-treated is brought into contact with or close to a heating body **28** at high temperatures, thereby being heated." Ex. 1023 at 7:6-11.

Matsutani Figure 4e



A small hole **30** through which a work piece (wire) can be inserted is made in a heating pod **29**, and only a portion 1A to be heat-treated is inserted into this small hole **30**, thereby being partially heated. Here, the portion not to be inserted into the heating pod **29** (portion not to be heat-treated) **1B** may be blown by cool air, thereby being actively cooled." Ex. 1023 at 7:13-19.

177. In my opinion, one of ordinary skill in the art would recognize that the five methods taught by Matsutani for partially heat-treating files are not difficult or expensive. The first method requires a heat gun and a water bath. The second method requires an insulating material and a furnace. The third method requires a salt bath. The fourth method requires a heating element and water. And the fifth

method requires a heating element. Ex. 1023 at 6:54 - 7:19. These methods require basic heating and cooling equipment and techniques. Accordingly, based on the advantages that Matsutani describes for his methods, it is my opinion that one of ordinary skill in the art would have no reason to change the heating method or equipment.

178. In my opinion, one of ordinary skill in the art would not discredit and ignore the fact that Matsutani teaches selective, partial heat-treatment of the tip to overcome specific problems related to the use of the file in a root canal therapy. In my opinion, a person of ordinary skill in the art would find that Matsutani's process is distinct from the process claimed in claims 1-17 of the '773 patent, which heat treats the entire instrument at a temperature from 400°C up to the melting point of the alloy.

179. Further, as noted above, even if a person skilled in the art had heated the entire endodontic file such that the A_f temperature was greater than 37°C, it does not follow that the file is necessarily permanently deformable. Both Pelton and Kuhn—Petitioner's own cited references—show that even when nickel titanium is below its A_f (*i.e.*, shape recovery temperature) it still may have superelastic properties. Ex. 1006 at Fig. 5 (see 0°C and 10°C stress strain curves); Ex. 1019 (unheated new ProFile with about 36°C A_f was superelastic at room temperature). Moreover, there is no evidence that the combination with an A_f greater than body

temperature would result in a file that exhibits the claimed amount of permanent deformation after the ISO 3630-1 bend test—greater than 10 degrees. I note that Dr. Goldberg was unaware of whether 5, 8 or 9 degrees of residual deformation were sufficient to preform *(i.e.*, pre-curve) an endodontic file. Ex. 2034 at 36:10-37:5. It is my opinion that Dr. Goldberg has not established (with any evidence) that the reference to "pre-curve" in Matsutani indicates the file would satisfy the permanent deformation requirement in the claims of the '773 patent.

I declare under penalty of perjury that the foregoing is true and correct. Executed this 2 day of November, 2015.

Robert Sinclair, Ph.D.

Exhibit A

ROBERT SINCLAIR
Professor

Department of Materials Science and Engineering
Stanford University
Stanford, California 94305-4034

Telephone: 650-723-1102
FAX: 650-725-4034
E-mail: bobsinc@stanford.edu

EDUCATION

B.A.	Materials Science	Cambridge University	1968
Ph. D., M.A.	Materials Science	Cambridge University	1972

CAREER

Permanent Positions

University of Newcastle-upon-Tyne, U.K.	Research Associate	1971 – 73
University of California, Berkeley	Research Engineer	1973 – 77
Stanford University		
Department of Materials Science and Engineering	Assistant Professor	1977 – 80
	Associate Professor (with tenure)	1980 – 84
	Professor	1984 – present
	Chair	2004 – 14
	Charles M. Pigott Professor in the School of Engineering	2009 – present
Stanford Nanocharacterization Laboratory	Director	2003 – 13
Bing Overseas Studies Program	Director	2010 – 12
Wallenberg Research Link	Associate Director	2013 – present

Visiting Positions

Centre d'Etudes Nucleaires, Grenoble	1981
HREM Laboratory, Cambridge University	1985
Matsushita Electric Industrial Company, Osaka	1987, 1989, 1991 – 94
Stanford Center for Japanese Studies, Kyoto	1997
Stanford Center in Oxford, U.K.	2001, 2010
Adjunct Advisor for International Center for Young Scientists, NIMS, Tsukuba	2004
Visiting Professor in the World Class University Project, Seoul National University, Korea	2008 – 2010

AWARDS

First Prize, Modern Metallography Micrograph Competition, 1972
First Prize Physical Science, Electron Microscopy Society of America Scientific Exhibit, 1975, 1976
Robert Lansing Hardy Gold Medal, The Metallurgical Society of AIME, 1976
Eli Franklin Burton Award, Electron Microscopy Society of America, 1977
Alfred P. Sloan Foundation Fellowship, 1979
Marcus E. Grossman Award, American Society for Metals, 1982
Excellence in Undergraduate Teaching, Stanford University Society for Black Scientists and Engineers, 1984, Stanford Society of Chicano/Latino Engineers and Scientists, 1993
Yamaha Prize for Best Poster, 1st International Conference on Metallic Multilayers, Kyoto, 1993
Fellow, (inaugural class) Microscopy Society of America, 2009
Distinguished Scientist, Physical Sciences, Microscopy Society of America, 2009
David Turnbull Lectureship Award, Materials Research Society, 2012
John M. Cowley Distinguished Lecture, Arizona State University, 2015

SELECTED PROFESSIONAL ACTIVITIES

Electron Microscopy Society of America: Program Committee, 1979, 1980, 1990, 1993; Bulletin Editorial Board, 1975-1981; Northern California Society President, 1980; Executive Committee, 1979-1982; Twelfth International Congress (ICEM) Program Committee, 1988-1990; Fourteenth ICEM Program Committee, 1997-1998; Fifteenth ICEM Program Committee, 2001-2002.

The Metallurgical Society of AIME: Alloy Phases Committee, 1977-1992; Northern California Section Chairman, 1979.

Materials Research Society: Symposium Organizer, 1987, 1990, 1994, 1995, 2010.

Organizer: Stanford Symposium on Applications of Contemporary Electron Microscopy, 1979-1994.

Organizer: US-France Asilomar Symposium on Amorphous Nickel-Titanium 1987.

A.S.U. National HREM Facility: Outside Proposal Review Committee, 1982-1990; Winter School Instructor, 1982-2006, 2011.

Electron Microscopy Short Courses: Harbin Institute of Technology, China, 1980; Linkoping University, Sweden, 1980, 1985; Korea Advanced Institute of Science and Technology, Seoul, 1988; Samsung Advanced Institute of Technology, Suwon, Korea, 2001, 2002.

Stanford University Short Course for Philips Technicians, 1989-1992, 1994.

Editorial Board: Journal of Applied Physics, 1994-1996; Journal of Electron Microscopy, 1996-present; and other journals.

Organizer: U.S.–Japan Workshop on "The Contributions of *In Situ* Electron Microscopy to the Understanding and Creation of Advanced Materials", Stanford Japan Center, Kyoto, Japan, 2000.

Co-Organizer: United Engineering Foundation Conference on Nanostructured Magnetic Materials, Irsee, Germany, 2002.

Co-Organizer: 70th Birthday Symposium in honor of Professor Gareth Thomas, ICEM XV, Durban, South Africa, 2002.

Organizer: In Situ Electron Microscopy Symposium, ICEM XV, Durban, South Africa, 2002.

Co-Organizer: Symposium on Semiconductor and Magnetic Materials, Microscopy and Microanalysis Conference, Quebec, Canada, 2003.

Chair: National Research Council Committee on Smaller Facilities, 2003-2006.

Organizer: ECI Conference: “Innovative Dynamic Studies of Materials at the Nanoscale”, Gyeong-ju, Korea, 2008

Co-Organizer: “International Workshop on Remote Electron Microscopy for *In Situ* Studies”: Stanford University, Stanford, California, 2008; Chalmers University of Technology, Gothenburg, Sweden, 2009; Carnegie Mellon University, Pittsburg, Pennsylvania, 2011.

Organizer: Symposium on Medical Applications of Nanotechnology, 11th International Congress on Advanced Materials, Rio de Janeiro, Brazil, 2009.

Co-Organizer: Symposium on Microscopy of Biological, Biomimetic and Medical Materials, 17th International Microscopy Congress, Rio de Janeiro, Brazil, 2010.

Co-Organizer: Symposium on Nanofunctional Materials, Nanostructures, and Nanodevices for Biomedical Applications II, Materials Research Society Fall 2010 Meeting, Boston, Massachusetts, 2010.

Co-Organizer: Stanford – Chalmers Workshop on Advancing Materials Innovatively, Stanford, California, 2012

Committee Member: National Science Foundation Materials 2022 Committee, 2012

Co-Organizer: Symposium on Nanoparticles: Applications and Bio-safety Issues, 18th International Microscopy Congress Prague, 2014

Co-Organizer: IAMNano 2014, International Workshop on Advanced and In-situ Microscopies of Functional Nanomaterials and Devices, Rio de Janeiro, 2014

COURSES TAUGHT (*present offerings)

Atomic Arrangements in Solids (G1); X-ray Diffraction Laboratory (UG1-4); Transmission Electron Microscopy (G2+)*; Nano-Characterization of Materials (G2+)*; Nanostructure and Characterization (UG1-4)*; Imperfections in Crystalline Solids (G1); Introduction to Materials Science: Nanotechnology

Emphasis (UG1-4)*; Microscopic World of Technology (UG1-4); Japanese Companies and Japanese Society (UG2)*; Infrastructure of British Science and Technology (UG 3); Soccer and English Society (UG 3)

RESEARCH INTERESTS

Structure-property-processing correlations in materials, using high-resolution microscopy and diffraction techniques, application to development of integrated circuit and magnetic recording materials. Introduction of in situ high resolution electron microscopy.

PUBLICATIONS

1 NAS government report, 5 edited works, 6 book chapters, 238 archival journal articles, 204 conference articles, 2 patents. (List available on request).