## American Journal of ORTHODONTICS and DENTOFACIAL ORTHOPEDICS

Founded in 1915

Volume 90 Number 1 July, 1986

Copyright © 1986 by The C. V. Mosby Company

### ORIGINAL ARTICLES

## The super-elastic property of the Japanese NiTi alloy wire for use in orthodontics

Fujio Miura, D.D.S., D.M.Sc.,\* Masakuni Mogi, D.D.S., D.D.Sc.,\*\* Yoshiaki Ohura, D.D.S.,\*\*\* and Hitoshi Hamanaka, Eng. Dr.\*\*\*\*

Tokyo, Japan

A new Japanese nickel-titanium (NiTi) alloy wire was developed by the Furukawa Electric Co., Ltd. of Japan. This wire was subjected to uniaxial tensile testing and a specially designed three-point bending test to determine the wire stiffness, and to evaluate springback, shape memory, and superelasticity. The Japanese NiTi wire exhibited an unusual property termed "super-elasticity," which no other orthodontic wire has shown. This phenomenon was researched thoroughly. The wire delivered a constant force over an extended portion of the deactivation range. Among all the wires compared, Japanese NiTi alloy wire was the least likely to undergo permanent deformation during activation. The new alloy exhibited a specific stress-strain curve unlike those of the other tested materials. Stress remained nearly constant despite the strain change within a specific range. This unique feature is the manifestation of so-called super-elasticity. Heat treatment enabled the load magnitude at which super-elasticity is reflected to be influenced and controlled by both temperature and time. A unique and useful process was also developed so that an arch wire delivering various magnitudes of force for a given activation could be fabricated from the wire of the same diameter. The clinical application of wires of this new alloy should be more likely to generate a physiologic tooth movement because of the relatively constant force delivered for a long period of time during the deactivation of the wire. Japanese NiTi alloy should be considered an important material addition to clinical orthodontic metallurgy. (AM J ORTHOD DENTOFAC ORTHOP 90: 1-10, 1986.)

Key words: NiTi alloy, super-elasticity, shape memory, arch wires

N ickel titanium (NiTi) alloy was investigated and developed by the Naval Ordinance Laboratory in Silver Springs, Maryland, in the early 1960s. It has also been reported that this alloy has a unique property called shape memory.

Shape memory is a phenomenon occurring in the alloy that is soft and readily amenable to change in shape at a low temperature, but it can easily be re-

From Tokyo Medical and Dental University.

DOCKE

\*Professor and Chairman, 1st Department of Orthodontics, School of Dentistry.

formed to its original configuration when it is heated to a suitable transition temperature.<sup>1-3</sup>

The shape-memory attributes of this alloy have attracted the attention and research endeavors of metallurgists in many countries. As a result it has been established that the NiTi alloy has excellent springback and super-elastic properties<sup>4,5</sup>; it has also been demonstrated that it has a high degree of resistance to corrosion.

The "super-elastic property" is a phenomenon that can be described briefly. The stress value remains fairly constant up to a certain point of wire deformation. At the same time, when the wire deformation rebounds, the stress value again remains fairly constant. Because

Find authenticated court documents without watermarks at docketalarm.com.

<sup>\*\*</sup>Lecturer, 1st Department of Orthodontics, School of Dentistry. \*\*\*Instructor, 1st Department of Orthodontics, School of Dentistry.

<sup>\*\*\*\*\*</sup>Associate Professor, Division of Metallurgy, Institute for Medical and Dental Engineering.

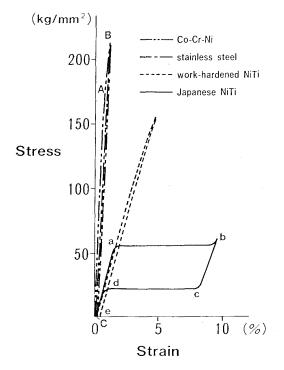


Fig. 1. Stress-strain curves for orthodontic wires of 0.016 inch in diameter at the moment of pulling and upon tensile release.

of such unique properties, NiTi alloy has been widely used in the industrial, medical, and other scientific fields.<sup>2,6</sup>

In clinical orthodontics, Andreasen and his associates were attracted by the unique properties inherent in NiTi alloy, such as the high elastic limit and the low elastic modulus. In 1971, they reported the results of their investigation for clinical use.<sup>7</sup> Subsequently, Unitek Corporation has produced this wire for the profession under the trade name of Nitinol. Nitinol has an excellent springback property,<sup>8-11</sup> but it does not possess shape memory or super-elasticity because it has been manufactured by a work-hardening process.<sup>2</sup>

In 1978, Furukawa Electric Co., Ltd. of Japan produced a new type of the Japanese NiTi alloy, possessing all three properties (excellent springback, shape memory, and super-elasticity).<sup>12-15</sup> The unique feature of the stress value remaining fairly constant during deformation and rebound is a very important concept of this entire investigation. Basic research was needed to study the Japanese NiTi alloy so that it might be used in clinical orthodontics to take advantage of the two unique properties of super-elasticity and shape memory.<sup>12-17</sup>

To thoroughly understand the super-elastic property

of Japanese NiTi alloy wire, the major thrust of this report is focused on the following aspects.

- 1. Examination of the mechanical property of the wire a. Tensile tests
  - b. Bending tests
- 2. Measurements of the influence of special heat treatment on the wire

### METHOD AND FINDINGS

### Examination of the mechanical property of the wire

*Tensile tests*. Uniaxial tensile testing was performed first because it is the most acceptable method to demonstrate clearly the comparative mechanical properties of wires.

The tensile strength was tested with the wire specimen attached to a steel plate with epoxy resin at  $37^{\circ} \pm 1^{\circ}$  C. Fig. 1 indicates the stress-strain curve. Comparison was made with other wire specimens such as stainless steel,\* Co-Cr-Ni,† and Nitinol.‡ A specimen of the Chinese NiTi wire reported by Burstone was not available for inclusion in this test.<sup>22</sup> Four separate types of round wire 0.016 inch in diameter were selected. The elastic modulus was  $17-20 \times 10^3$ kg/mm<sup>2</sup> for the stainless steel wire and  $17-22 \times 10^3$ for Co-Cr-Ni. The elastic modulus of Nitinol wire was  $5-6 \times 10^3$  kg/mm<sup>2</sup>, showing the stress-strain curve to be almost straight. In contrast, a stress-strain curve of great significance was produced with the Japanese NiTi alloy wire, yielding a significantly higher value of elastic modulus than the Nitinol wire. When the stretch exceeded 2%, the stress value was not changed appreciably. When the strain was induced at 8%, it produced stresses of 55 to 58 kg/mm<sup>2</sup>. When the wire specimen was then stretched 8% or more, the stress was increased further. This unusual property of the Japanese NiTi alloy wire as illustrated by the stress-strain curve is called "super-elastic property" (Fig. 1, a-b).

When strain was reduced, the stainless steel, Co-Cr-Ni, and Nitinol wires all exhibited almost straight stress-strain curves. In comparison, when strain was reduced, the Japanese NiTi alloy wire did not change proportionally to the stress decrease from 8% to 2%. There was no permanent set when the stress reached zero. This is also called "super-elastic property" (Fig. 1, c-d). The portion representing the superelasticity was made much lower by decreasing the stress rather than by increasing it.

Bending tests. To determine the possible use of the super-elastic property in clinical orthodontics, a three-

<sup>\*</sup>Tru-chrome, Rocky Mountain/Orthodontics, Denver, Colo. †Elgiloy, Rocky Mountain/Orthodontics, Denver, Colo. ‡Unitek Corporation, Monrovia, Calif.



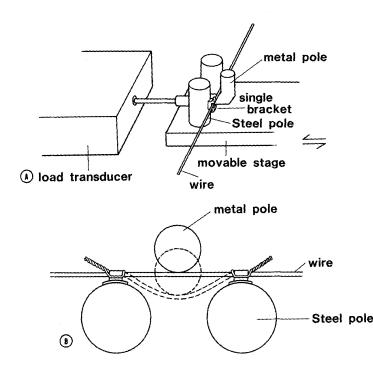


Fig. 2. Schematic drawing of instrumentation used in the three-point bending test. A, Side view. B, View from above.

point bending test was conducted in a specially designed situation similar to the conditions involved in moving teeth in the oral cavity.

The approved ADA standard method is a cantilever type of test. This method is an acceptable one to demonstrate the springback properties. Wires of good springback property increase the length and the angle of the specimen so that the super-elastic-like property appears even if the wires do not possess this feature.

As a result the ADA standard test method was not used in this investigation. Instead, a three-point bending test was designed because this would accurately differentiate the wires that do not possess super-elastic features. At the same time the three-point bending test actually simulates the application of wire pressure on the teeth in the oral cavity.

A three-point bending test was conducted by designing a bend test instrument as shown in Fig. 2. This was designed to clarify the relationship between the loading and deflection by determining the nature of the force being delivered during orthodontic treatment. A single bracket was attached on a steel pole, acting like a dental unit placed on a movable stage, so that the bracket span could be set at 14 mm. The test wire was held in place with a ligature wire in the slot with a known quantity of force. The midportion of the wire segment was then deflected 2 mm at the speed of 0.1 mm/min under the pressure from a metal pole 5 mm in diameter.

Fig. 3 shows the results of the bending test with several different types of 0.016 inch wires. The load deflection curves represented by both stainless steel and Co-Cr-Ni wires showed a linear relationship up to 0.7 mm. When it exceeded 0.7 mm, its increasing ratio was gradually reduced. When the amount of deflection was 2.0 mm, the load was 1320 to 1370 g, and on reducing the deflection to 1.8 mm, the load decreased rapidly. The subsequent decreasing ratio was proportional and the permanent deformation was 0.65 mm. The dotted line in Fig. 3 represents Nitinol, which indicates the applied load moment for the corresponding deflection of up to 2 mm. The load deflection curve represented by Nitinol was almost linear along with the additional loading. When the deflection distance of 2.0 mm was reached, the load was 790 g. As the deflection was removed, the load was decreased in a similar manner as indicated by the stainless steel and Co-Cr-Ni wires. Fig. 3 shows the load deflection curve of the Japanese NiTi alloy wire to be almost linear up to 0.7 mm. The load-increasing ratio was decreased, and the load was 650 g when the deflection was 2.0 mm. During the decrease of the deflection, the load decreased rap4 Miura et al.

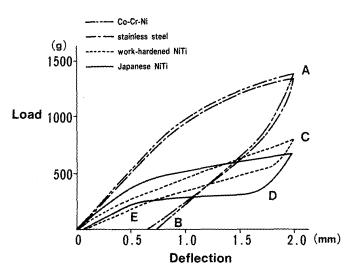


Fig. 3. Load deflection curves produced by the three-point bending test.

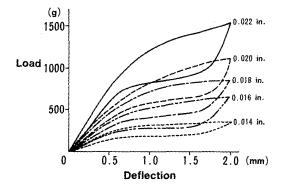


Fig. 4. Load deflection curves on five different diameters of the Japanese NiTi alloy wires.

idly in the range of the deflection curve between 2.0 mm and 1.6 mm. When the deflection was decreased 1 mm from 1.6 mm to 0.6 mm, the load was decreased only a small amount within the range of 250 to 350 g. When the deflection was less than 0.6 mm, the load was decreased within a given proportion and the permanent set was only 0.01 mm. By evaluating the test results, both the bending test and the tensile test demonstrated that Japanese NiTi alloy wire possesses super-elastic property.

Fig. 4 shows the result of the bending test of five different diameters—0.014, 0.016, 0.018, 0.020, and 0.022 inch—of the Japanese NiTi alloy wires using the same testing instrument. The diagram illustrates the load needed (vertical component) to produce the deflections of up to 2 mm for the selected gauges.

# Measurement of the influence of special heat treatment on the wire

Heat treatment of NiTi alloy does make a dramatic change in its mechanical property.23 To attain optimal use of the super-elastic property in clinical orthodontics, the influence of a varied series of heat treatments was studied. A comparative analysis was conducted for this property before and after being subjected to heat using a 0.016 inch Japanese NiTi alloy wire. Heat treatment was produced by immersion of the wire in a nitrate salt bath. The temperature levels applied to the wire were 200° C, 300° C, 400° C, 500° C, and 600° C, respectively. The heat exposure periods were 5, 10, 60, and 120 minutes. A total of 20 different variations of heat treatment were used. After the heat exposure, the wire was quenched in water. The mechanical properties of the wire were then determined by conducting a series of bending tests on the bend test instrument (Fig. 2). The test was conducted at a temperature of  $37^{\circ} \pm 1^{\circ} C$ .

No significant change of mechanical property of wire was noted at  $200^{\circ}$  C or  $300^{\circ}$  C. Figs. 5 through 7 show the results of the bending test at temperatures of  $400^{\circ}$  C,  $500^{\circ}$  C, and  $600^{\circ}$  C, respectively. Fig. 5 shows the results of the heat application at  $400^{\circ}$  C. The portion indicating the linear relationship between the load and the deflection showed that only a small amount of heat treatment effect was noted. In the portion showing super-elasticity, the load value was reduced along with the lapse of time.

In Fig. 6, at 500° C temperature, the load portion

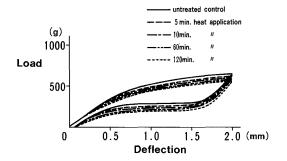


Fig. 5. Results of the heat application on 0.016 inch Japanese NiTi alloy wire at 400° C.

showing super-elasticity was definitely decreased. The decrease of the load was greater than at 400° C (Fig. 5). The wire, treated at 500° C for 120 minutes, had a super-elastic portion of approximately 50 g load along with the gradual removal of load. The super-elastic load portion decreased considerably in comparison to the load of 300 g in the untreated control wire (Figs. 3 and 4).

Fig. 7 indicates the results after heat application at  $600^{\circ}$  C. Super-elasticity and the good springback property of the wire were almost completely lost even when the heat exposure was for only 5 minutes.

### DISCUSSION

#### **Evaluation of findings**

Tensile tests. This basic test was conducted to analyze the mechanical properties of the wire. As seen in Fig. 1, the stress-strain curves of stainless steel and Co-Cr-Ni alloys, indicated as 0-A, demonstrated elastic deformation. This revealed that the relationship between stress and strain was proportional. When the stress was increased, as indicated by A-B, permanent deformation was produced. In other words, the stress increase was disproportional to the strain increase. Subsequently, when the stress was removed, the relationship between the stress and the strain was almost proportional and permanent deformation remained as indicated by C-0.

Nitinol, the work-hardened NiTi alloy wire, possessed a much lower elastic modulus and could be deformed almost five times greater, but the pattern of stress–strain curve was quite similar to that of the stainless steel and Co-Cr-Ni wires.

The Japanese NiTi alloy wire produced a completely different curve pattern as shown in Fig. 1. When the wire was stretched up to 2%, as indicated by 0-a, the stress and strain were almost proportional. As indicated

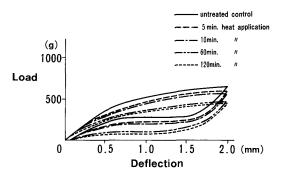


Fig. 6. Results of the heat application on 0.016 inch Japanese NiTi alloy wire at 500° C.

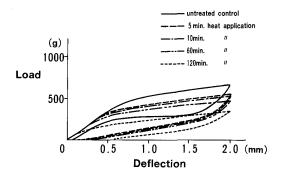


Fig. 7. Results of the heat application on 0.016 inch Japanese NiTi alloy wire at 600° C.

by a-b, the stress was not subsequently changed in spite of the strain increase. This appeared to be similar to the curve shown by the plastic deformation, but when the stress was removed, the phenomenon indicated by c-d was similar to a-b. This phenomenon is the superelastic property of this alloy, which shows a completely different property compared with the other wire samples.

The physical behavior of the NiTi alloy wire can be interpreted and explained from a metallurgic analysis. It is a generally accepted fact that NiTi alloy is a nearly equi-atomic intermetallic compound that incorporates a variety of properties that can be controlled by the manufacturing method. A given zone lies between high and low temperature ranges. At the high temperature range, the crystal structure of NiTi alloy is in an austenite phase, which is a body-centered cubic lattice (CsCl type B2 structure). The martensitic phase, which is a close-packed hexagonal lattice, is at a low temperature range. By controlling the low and high temperature ranges, a change in crystal structure called martensitic transformation can be produced. This phe-

Volume 90 Number 1

# DOCKET A L A R M



# Explore Litigation Insights

Docket Alarm provides insights to develop a more informed litigation strategy and the peace of mind of knowing you're on top of things.

# **Real-Time Litigation Alerts**



Keep your litigation team up-to-date with **real-time alerts** and advanced team management tools built for the enterprise, all while greatly reducing PACER spend.

Our comprehensive service means we can handle Federal, State, and Administrative courts across the country.

## **Advanced Docket Research**



With over 230 million records, Docket Alarm's cloud-native docket research platform finds what other services can't. Coverage includes Federal, State, plus PTAB, TTAB, ITC and NLRB decisions, all in one place.

Identify arguments that have been successful in the past with full text, pinpoint searching. Link to case law cited within any court document via Fastcase.

# **Analytics At Your Fingertips**



Learn what happened the last time a particular judge, opposing counsel or company faced cases similar to yours.

Advanced out-of-the-box PTAB and TTAB analytics are always at your fingertips.

## API

Docket Alarm offers a powerful API (application programming interface) to developers that want to integrate case filings into their apps.

### LAW FIRMS

Build custom dashboards for your attorneys and clients with live data direct from the court.

Automate many repetitive legal tasks like conflict checks, document management, and marketing.

### FINANCIAL INSTITUTIONS

Litigation and bankruptcy checks for companies and debtors.

### E-DISCOVERY AND LEGAL VENDORS

Sync your system to PACER to automate legal marketing.