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# The Effect of Pre-Stressing on the Static Indentation Load Capacity of the Superelastic 60NiTi

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#### Abstract

Superelastic nickel-titanium alloys, such as 60NiTi (60Ni-40Ti by wt.%), are under development for use in mechanical components like rolling element bearings and gears. Compared to traditional bearing steels, these intermetallic alloys, when properly heat-treated, are hard but exhibit much lower elastic modulus (~100 GPa) and a much broader elastic deformation range (~3 percent or more). These material characteristics lead to high indentation static load capacity, which is important for certain applications especially space mechanisms. To ensure the maximum degree of elastic behavior, superelastic materials must be pre-stressed, a process referred to as "training" in shape memory effect (SME) terminology, at loads and stresses beyond expected use conditions. In this paper, static indentation load capacity tests are employed to assess the effects of pre-stressing on elastic response behavior of 60NiTi. The static load capacity is measured by pressing 12.7 mm diameter ceramic  $Si_3N_4$  balls into highly polished, hardened 60NiTi flat plates that have previously been exposed to varying levels of pre-stress (up to 2.7 GPa) to determine the load that results in shallow but measurable (0.6 µm, 25 µin, deep) permanent dents. Hertz stress calculations are used to estimate contact stress. Without exposure to pre-stress, the 60NiTi surface can withstand an approximately 3400 kN load before significant denting (>0.4 µm deep) occurs. When pre-stressed to 2.7 GPa, a static load of 4900 kN is required to achieve a comparable dent, a 30 percent increase. These results suggest that stressing contact surfaces prior to use enhances the static indentation load capacity of the superelastic 60NiTi. This approach may be adaptable to the engineering and manufacture of highly resilient mechanical components such as rolling element bearings.

#### Nomenclature

- HRC Rockwell C hardness
- E Young's modulus of elasticity, GPa
- $\sigma$  stress, GPa
- $\sigma_v$  yield stress, GPa
- $\delta, \Delta$  deflection, elongation
- ε strain
- v Poisson's ratio
- W load, Kg<sub>f</sub>
- F force, N
- D indenter diameter, mm
- dp dent depth, µm
- dp/D dent depth ratio

# Introduction

The nickel-rich, binary nickel-titanium alloy 60NiTi is among several intermetallic, superelastic alloys currently under consideration as candidate materials for use in mechanical components (Ref. 1). Materials such as 60NiTi (60Ni-40Ti by wt%) exhibit an unusual combination of physical properties that impact the design and performance of mechanical components especially rolling element bearings (Ref. 2). Namely, 60NiTi can be hardened to high levels (Rockwell C 58-62), has a relatively low apparent elastic modulus (E~100 GPa) and exhibits a very large elastic deformation range (typically >3 percent). The combination of these three physical characteristics impart extraordinarily high resiliency that translates to high static indentation load capacity, an important attribute for a bearing material. Additionally, because the composition contains no iron, such alloys are immune to atmospheric rusting behavior observed even for the stainless steel 440C. Table I gives the approximate properties of 60NiTi along with the shape memory alloy 55NiTi and selected conventional bearing materials.

Property	60NiTi	55NiTi	440C	Si <sub>3</sub> N <sub>4</sub>	M-50
Density	6.7 g/cc	6.5 g/cc	7.7 g/cc	3.2 g/cc	8.0 g/cc
Hardness	56-62 HRC	35-40 HRC	58-62 HRC	1300-1500 Hv	60-65 HRC
Thermal conductivity W/m-°K	18	19	24	33	~36
Thermal expansion	~12.4×10–6/°C	~10×10–6/°C	10×10–6/°C	2.6×10–6	~11×10–6/°C
Magnetic	Non	Non	Magnetic	Non	Magnetic
Corrosion resistance	Excellent	Excellent	Marginal	Excellent	Poor
Tensile/flexural strength	~1000 MPa	~900 MPa	1900 MPa	600-1200 MPa (bend strength)	2500 MPa
Young's Modulus	~95 GPa	~100 GPa	200 GPa	310 GPa	210 GPa
Poisson's ratio	~0.34	~0.34	0.3	0.27	0.30
Fracture toughness	TBD	TBD	22 MPa/√m	5-7 MPa/√m	20-23 MPa/√m
Maximum use temp	~500 °C	~300 °C	~400 °C	~1100 °C	~400 °C
Electrical resistivity	$\sim 80 \times 10^{-6} \Omega$ -cm	$\sim 80 \times 10^{-6} \Omega$ -cm	$\sim 36 \times 10^{-6} \Omega$ -cm	Insulator	$\sim 60 \times 10^{-6} \Omega$ -cm

TABLE L-	-NOMINAL	PROPERTIES FOR	R CONVENTIONAL	BEARING ALLOYS	AND 55NiTi AND 60NiTi
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TBD: "to be determined"

The high static indentation load capacity was first verified experimentally in an earlier study by the authors (Ref. 2). The primary impetus for that work was derived from data obtained from rather conventional compressive strength tests conducted as part of a more general superelastic materials characterization program. In these tests, small, right cylinders of hardened 60NiTi were compressed in a load frame to generate a stress-strain curve. Tests of traditional bearing steels give expected deformation behavior in the form of the well-known "stress-strain curve, initiating at the origin and climbing in a straight line following Hooke's law of elastic deformation (Ref. 3). Figure 1 shows such a plot for the bearing steels 440C and 52100 and the high carbide tool steel REX20 using data derived from the literature (Ref. 4). For comparison, the curve for the structural titanium alloy Ti-6V-4Al that has an elastic modulus close to that estimated for NiTi, is also shown.

The slope of the line in the linear region is used to estimate the elastic modulus. The transition point from linear to nonlinearity signifies that the elastic strength limit has been reached and that permanent plastic deformation has begun. For common engineering metals, the yield strength is estimated to be the point on the curve where a line drawn parallel to the linear region but offset by a strain of 0.2 percent intersects the curve. This stress limit ( $\sigma_v$ ) is then used with suitable engineering margin in the design



Figure 1.—Approximation of the elastic behavior of bearing steels compared to titanium. Data adapted from Reference 4.

process. Such measurements have been made for many decades and one can grasp the basic behavior of a material based upon the nature of the plot. Highly ductile metals like aluminum, for instance, have stress-strain plots similar to that of titanium alloys except that they reach their elastic limit much earlier and exhibit a broad, extended plastic strain range. Brittle materials have steep slopes and exhibit very small elastic range with little or no plastic behavior. Rather, for brittle materials the plot ends abruptly at failure at the maximum extent of the linear region.

Figure 2 shows the stress-strain plot for 60NiTi and includes the data from the very first compression test and subsequent unloading and reloading cycles on the same specimen. Several similarities and differences are observed that contrast the behavior of traditional metals like titanium and conventional hard bearing steels.

One feature of the compression stress-strain behavior of 60NiTi is that it is not exactly linear, that is, the elastic modulus is apparently not constant. During compression, it appears that the modulus is a bit higher at low stresses and strains and gradually decreases as the strain and stress levels rise. Another unique characteristic is that the strain that occurs during the very first compression cycle is not fully recovered when the load is removed. A permanent deformation offset of nearly 1 percent remains at the end of the first cycle. Subsequent cycles show fully recoverable strain and a continuation of a somewhat non-linear elastic modulus. In addition, it appears that 60NiTi exhibits a small amount of hysteresis, meaning that the strain during the unload portion of the cycle lags the stress resulting in a loop shaped plot depicted in Figure 2 by the green lines. Though small in magnitude, this hysteresis is sometimes associated with internal material damping. If one considers the repeatable elastic response of later test cycles from Figure 2 an averaged stress strain curve can be generated. It is plotted schematically in Figure 3 along with the curves from Figure 1 for comparative purposes.



Figure 2.—Compressive elastic behavior of 60NiTi. Dashed line represents initial compression cycle. Green solid lines represent subsequent compression-relaxation cycles. Data chart from Reference 2.



Figure 3.—Estimated and measured averaged elastic stress-strain behavior of 60NiTi compared to bearing steels and titanium alloy.

Referring again to the elastic compression behavior of 60NiTi depicted in Figure 2, perhaps of more significance than the shape of the curve, is the magnitude of the elastic strain. Following the initial load-

unload cycle in this test, 60NiTi exhibits fully recoverable strains of around 5 percent. In fact, 60NiTi may well be able to endure even higher fully elastic strains than shown here. The load frame fixtures used, not the material capability of the 60NiTi test specimens, limited the stress level (Ref. 2). Compared to the maximum elastic strain capability of conventional bearing steels (~1 percent) it is clear that 60NiTi is far more elastic. This extensive elastic deformation range combined with high hardness and a relatively low elastic modulus results in a high resiliency, static load capacity and tolerance to indentation damage.

This combination effect was shown conclusively for 60NiTi and has been reported (Ref. 2). That work involved loading large diameter (12.7 mm) hard ceramic spheres (Si<sub>3</sub>N<sub>4</sub>) onto highly polished 60NiTi surfaces that had been heat treated to achieve a Rockwell hardness comparable to conventional bearing steels (HRC 58-62). Figure 4 shows the results of those tests in which the data is plotted as permanent dent depth (dp) as a function of static load. The horizontal line set at a permanent dent depth of ~0.6 µm represents the generally accepted damage limit for a quiet running bearing where dents shallower than the indenter diameter multiplied by  $5 \times 10^{-4}$  can be tolerated. Also shown in Figure 4 are the discrete static load limits for other bearing materials. If considered solely on the basis of static load capacity 60NiTi is robust. Of course, there are many other engineering and material properties that must be weighed when selecting a bearing material.

One important material characteristic to consider when manufacturing a precision component like a bearing is dimensional stability. Since 60NiTi belongs to the family of superelastic materials that include the shape memory alloys, static dimensional stability especially over a wide temperature range is a credible concern. For the NiTi alloy system, the addition of nickel beyond a stoichiometric ratio (i.e., more than 55 wt%) depresses any latent shape memory effects well below ambient conditions. This was shown in an earlier investigation (Ref. 1). At temperatures ranging from near absolute zero to over 500 °C, 60NiTi is dimensionally stable except for ordinary, smooth and predictable thermal expansion (Ref. 1).

Looking at the compression data in Figure 2 however, a dimensional problem does, in theory, exist. The potential problem is that during the very first loading cycle, permanent deformation of nearly 1 percent occurs. The exact reason for this initial loading cycle permanent deformation is not exactly known but it is likely related to atomic level phase structure and it is a phenomenon well recognized by the shape memory alloy community (Ref. 5). In shape memory effect material applications, components are exercised (strained) at loads and deflections beyond those expected in service. This pre-straining treatment is sometimes called training and results in more fully elastic and repeatable shape memory behavior. Since 60NiTi is in the superelastic shape memory family of materials, it seems a reasonable approach to use an analogous pre-straining process to assure more fully elastic behavior for bearings.





In the present investigation, we examine the efficacy of pre-stressing on 60NiTi surfaces to enhance their elastic response. We will use indentation experiments conducted on baseline and pre-stressed, highly polished 60NiTi surfaces to determine if the small but permanent deformation observed for the first loading cycle (shown in Figure 2) can be reduced or even eliminated. In effect, we will use compressive stress applied to the surface of hardened and polished 60NiTi to train the material so that subsequent loads applied will elicit a fully elastic response.

In a recent previous study, we developed specimens and a technique to measure the static indentation load capacity for as heat-treated 60NiTi (Ref. 6). In the following sections, this work will be extended to include measuring the indentation load capacity of 60NiTi surfaces after they have been statically prestressed at loads corresponding to stresses up to 2.7 GPa. Since near term applications for 60NiTi include space instrument bearings that have to endure high vibration shock loads during launch, it is imperative that the indentation load capacity behavior of pre-stressed material be characterized.

To study this phenomenon, specially designed 60NiTi indent specimens are prepared using an advanced pre-alloyed powder metallurgy process (Ref. 6). These specimens present a flat, highly polished surface to be indented with ceramic ( $Si_3N_4$ ) bearing quality balls in an effort to mimic the contacts inside a ball bearing. Prior to indentation, the polished surfaces are carefully pre-stressed (compressed) with a hard, flat-faced ceramic cylinder to reduce or eliminate non-elastic deformation. It is expected that these measurements will help elucidate the contact behavior of hard superelastic candidate bearing materials like 60NiTi.

### **Material Background and Test Procedures**

Buehler and his colleagues at the Naval Ordinance Laboratory first invented 60NiTi in the late 1950s as a high temperature missile nose cone alloy. They studied it intensively throughout the following decade until the early 1960s. Their research interests then changed for two primary reasons (Ref. 7). The first reason was their discovery of the shape memory effect (SME) of 55NiTi. The unusual SME provided rich opportunities for new technological developments and was thus of more interest than a simple structural alloy like 60NiTi. The second major impetus to abandon research on 60NiTi was that it was very difficult to work with and manufacture. 60NiTi was difficult to machine and was prone to spontaneous fracture especially during cooling from heat treatment temperatures (Ref. 8). Since that time, the development of modern powder metallurgy processing methods typically applied to ceramics and titanium materials has largely overcome both of these obstacles. 60NiTi can now be produced in a form that can be machined using conventional processes though as a relatively immature material, quality variability is still encountered (Ref. 6).

The indentation specimens tested are cut from ingots of 60NiTi made via hot isostatic pressing (HIP) of high purity pre-alloyed powders. The powder metallurgy process is designed to yield material with a uniform, fine-grained microstructure that can be readily machined prior to hardening and is largely free of flaws, inclusions and voids that can cause spontaneous fracture during quenching (Ref. 9). Figure 5 shows cross sections of the test material before and after a typical heat treatment that consists of a 1000 °C vacuum solution treatment followed by rapid water quench. This process results in typical hardness values of HRC 60-62.

Metallurgical cross sections of sample materials that were polished and acid etched revealed the unexpected presence of large ( $\sim$ 150 µm) unconsolidated 60NiTi particles within the overall matrix. Though still under investigation, the cause for the incomplete particle consolidation during the HIP phase of the material production appears to be surface oxidation of random discrete 60NiTi coming from the pre-alloying powder production step. Improving powder quality and assessment techniques are ongoing. With respect to the present study concerning static indentation load capacity, previous indentation experiments of 60NiTi with and without such flaws have shown that these large-scale features have no measurable effect on the indentation behavior except in the case where complete specimen fracture occurs (Ref. 6). In this paper, pre-stress levels and indentation loads are below fracture levels.



Figure 5.—Optical micrographs of 60NiTi made via the NASA-Abbott powder metallurgy process (a) before and (b) after heat treatment process. Cross sections have been polished and acid etched to accentuate microstructure.

The basic experimental set-up presents a highly polished 60NiTi surface to a ceramic  $(Si_3N_4)$  indenter ball. Prior to indentation, selected surfaces are pre-stressed by pressing with a flat, highly polished ceramic counter-face  $(Si_3N_4)$ . Achieving this arrangement in a convenient and practical manner was not straightforward.

To facilitate shallow dent location following testing, a unique plate geometry was developed that consists of a large, thick plate of 60NiTi with multiple separate small diameter raised regions (lands) that each serve as a site for a single indent experiment. This specimen configuration also made metallographic polishing of the test surfaces convenient. Figure 6 shows a photograph of the indent specimens.



Figure 6.—60NiTi indentation specimen made to facilitate metallographic polishing, indent location and to prevent indent events from confounding adjacent dent experiments.

The specimen size and shape closely matches standard metallographic mounts making polishing simple and convenient. Each specimen has nine circular (6 mm diameter) indent regions that stand proud of the specimen surface approximately 3 mm. One indent is positioned in the center of each of these polished raised lands. The overall specimen thickness (10 mm) and diameter (25 mm) conveniently fit inside the inner race of ball bearing blanks cut from larger ingots as part of a bearing development project.

The indentation testing is conducted in a modified load frame and the procedure mimics the standard Brinell test except that loads are kept below the level required to achieve a very deep and excessive depression. Figure 7 shows the test rig, the ball-plate contact and a representative dent impression. The general configuration and procedure is similar to that used by Park (Ref. 4) and his colleagues to establish the static load capacity of advanced, high carbide content steels and is described in more detail in the previous study by the authors (Ref. 6).

Prior to indentation testing, the surfaces of the raised lands were pre-stressed (compressed) using the same load frame set-up used for indenting. The main differences were that a highly polished, flat-faced,  $Si_3N_4$  ceramic cylinder replaces the Brinell indenter ball and the loads used were far higher. In Figure 8 below, a close-up photo shows a pre-stress experiment ready to begin.

To conduct a pre-stress, a computer-controlled program is used. First, the ceramic cylinder is manually brought to within 1 mm of the 60NiTi surface. Then the computer program is initiated and the cylinder is slowly lowered until contact is detected. The load is then ramped linearly over a 2-min period until the pre-stress load is reached. Once reached, the load is maintained for at least 1 min. Then the pre-stress load is removed. For the experiments presented in this paper, loads corresponding to two different stress levels (1.36, and 2.72 GPa) were used. Higher pre-stress levels were originally planned but during pre-stressing to higher loads spontaneous specimen fracture sometimes occurred. Since specimen availability was limited at this time, only two pre-stress levels were investigated. Higher pre-stress level experiments may be conducted in the future when more uniform raw material becomes more readily available. It is expected that the pre-stress levels tested (to 2.72 GPa) will enable the general effects to be assessed.



Figure 7.—Brinell test configuration used for indentation experiments to measure static load capacity.



Figure 8.—Close up view of pre-stress configuration. A flat, polished  $Si_3N_4$  cylinder (upper) is loaded onto polished, flat 60NiTi raised land to apply pre-stress load.

Following pre-stressing, the surfaces are then indented to determine the threshold load above which measurable, visible dents occur. Indentation is achieved by pressing a 12.7 mm diameter  $Si_3N_4$  ball onto a raised land surfaces previously compressed. The indent ball is held in a hemispherical recess machined into a standard steel push rod. A small volume of silicone vacuum grease is applied to the surface of the recess prior to inserting the ball to help secure the ball against gravity and to ease subsequent ball removal. Figure 9 shows a close-up photograph of the set-up used for an indentation experiment.

To conduct the indentation test, the selected pre-stressed, raised land is positioned directly beneath the ball and aligned by eye such that the indent occurs at or near the center of the raised land. The push rod-ball assembly is then manually lowered by pressing the "lower head" control button on the load frame controller. When the ball is within 1 mm of the surface, the control button is released and the operator then engages an automated computer controlled indent program that slowly lowers the ball until a load cell detects contact. The load is then increased linearly at approximately 100 kg<sub>f</sub> per minute until the desired static load is reached at which time the load is held steady (within ~2 percent) by the control system. After a dwell period (typically 1 min) the push rod-ball assembly is raised to remove the load and the test concludes.



Figure 9.—Close up view static load indentation configuration. Spherical, polished  $Si_3N_4$  bearing ball (12.7mm diameter) is loaded onto polished, flat 60NiTi raised land to apply indentation load.



Figure 10.—60NiTi surface after indent made by 12.7 mm diameter Si<sub>3</sub>N<sub>4</sub> ball loaded to 500 kg<sub>f</sub>. (Diameter of raised land is 6 mm).

The goal of these tests is to determine the effects, if any, of pre-stressing on the static load capacity of the 60NiTi. In an earlier study, the baseline static indentation load capacity was characterized using noncontact optical profilometry measurements that yielded information on dent depth like that shown in Figure 10 (Ref. 6). The baseline data is also summarized in Figure 4.

For studying the effects of pre-stressing 60NiTi the same measurement techniques are used but the test loads are confined to the region where dent depths on the order of 0.5 to 1.0  $\mu$ m (dp/D~0.00005) are anticipated. It is dents of this magnitude that are considered the damage threshold for bearings when tested with a 12.7 mm diameter indenter. Based upon some preliminary exploratory trials, the selected indentation load range was 250 to 550 kg<sub>f</sub>(3200 to 5400 N). The majority of testing was done for the highest level of pre-stress, 2.76 GPa due to the limited availability of specimens. Three repeat tests were typically done for each test load. The data presented is the average. Limiting the test points to this region of interest also makes the specimen fabrication and data measurement tasks more manageable. Future tests, particularly those using curved 60NiTi samples cut from bearing races, may expand the data region to include lower and higher loads.

# **Results and Discussions**

The indentation data is summarized in Table II using a nominal constant value of 100 GPa for the modulus of 60NiTi. Due to resource constraints (raw materials, specimen preparation time, etc.) and sporadic specimen fracture encountered during pre-stressing, experimental measurements on pre-stressed surfaces were limited. To maximize the research outcome, test loads were confined to loads expected to yield dents near the 0.6 µm depth threshold. In the future, more complete data sets may be generated.

The dent depth data are plotted in Figure 11 as a function of indent load. Data measurement uncertainty was below 0.03  $\mu$ m (1.0  $\mu$ in.) and scatter amongst repeat tests was of the same magnitude but slightly higher (~0.15  $\mu$ m) except for low loads (order 250 kg<sub>f</sub>) where dent detection was very difficult and scatter increased. At the lowest loads, some dent experiments yielded no measurable dents and others resulted in dents on the order of a few hundredths of a micron. For comparison to bearing operating condition, the quiet running dent criterion is depicted as a horizontal line.

	Q		- /			
		Dent depth,				
Indentation load,	μm (μin.) Pre-stress level					
Kg <sub>f</sub>						
	0.0 GPa	1.36 GPa	2.76 GPa			
0	0	N/A	N/A			
50	None detected	N/A	N/A			
100	None detected	N/A	N/A			
150	None detected	N/A	N/A			
200	None detected	N/A	N/A			
250	0.06 (2.5)	0.05 (2.0)	N/A			
300	0.29 (11.8)	0.18 (7.1)	N/A			
350	0.31 (12.1)	0.38 (15.0)	N/A			
375	0.41 (16.1)	0.48 (18.9)	N/A			
400	0.74 (29.1)	0.86 (33.9)	0.31 (12.1)			
450	1.36 (53.5)	N/A	0.42 (16.5)			
500	1.8 (72)	N/A	0.64 (25.2)			
550	N/A	N/A	0.79 (31.3)			

TABLE II.—INDENTATION DATA SUMMARY
[Si <sub>3</sub> N <sub>4</sub> ball (12.7 mm dia.) loaded against hardened (HRC 60.5) polished flat 60NiTi surface]
Oujet running dent depth limit: $0.6 \mu m (dp/D=0.00005)$

N/A indicates data not acquired





When considering the data, it is apparent that pre-stress levels of 1.36 GPa do not affect the indentation load-dent depth response. Samples of 60NiTi that were pre-stressed at 2.7 GPa exhibit an improved ability to withstand indentation type loads. On the graph in Figure 11 this is manifested as a shift of the data curve to a higher load level. The dent behaviors of conventional and high performance bearing steels are also plotted in the figure using the data from the literature (Refs. 6 and 7). It is clear that from a static load capacity consideration alone, the 60NiTi outperforms conventional bearing steels, even those like REX20 that are much harder. If one considers contact stress, however, the results differ.



Figure 12.—Indent depth as a function of mean contact stress for baseline and pre-stressed 60NiTi, plates loaded with a ~12.7 mm diameter  $Si_3N_4$  indenter ball. Dent depth data for 440C and REX20 are taken from Reference 7 are shown for comparison. 60NiTi's modulus assumed as 100 GPa.

Figure 12 plots the same dent depth data as a function of mean contact stress. Since the elastic modulus for 60NiTi is relatively low, during indentation by the stiff  $Si_3N_4$  balls more elastic deformation occurs than for the other bearing alloys. This effectively increases the contact area and reduces peak and mean contact stresses. This enables the 60NiTi superelastic surface to withstand higher loads than steels even though its contact stress limit seems to fall between that of 440C and REX20. With the exception of ceramics like  $Si_3N_4$ , bearing construction materials all have about the same modulus and thus the dent depth plots looked the same regardless of whether they were versus stress or load. For 60NiTi this is not the case. Given that bearing load capacity is measured in force, it appears that the use of 60NiTi allows much higher load levels before permanent deformation that affects performance occurs.

The reason or reasons why 60NiTi can withstand higher contact loads before significant permanent deformation occurs is an open subject under ongoing investigation. 60NiTi belongs to the family of superelastic materials that are known to exhibit large recoverable elastic strains especially in compression. In tension, 60NiTi as well as other intermetallic alloys tend to be brittle are more notch sensitive than steels. More common superelastics, like 55NiTi are relatively soft and exhibit shape memory behavior. In addition, shape memory alloys are often strained at stress levels beyond their expected use conditions to ensure repeatable and more fully recoverable strain-temperature response. Based upon the denting results generated in the current paper, it seems reasonable to infer that the 60NiTi is responding to pre-stressing in a manner similar to that of shape memory alloys responding to "training" to enhance the repeatability of their strain response. The primary difference being that shape memory alloys are conditioned to respond to temperature in a repeatable and superelastic manner. Pre-stressing of the hardened 60NiTi material conditions it to exhibit a fully elastic response to a stress level at least as high as the pre-stress condition.

While it might be elucidating to prepare metallurgical cross-section samples from the dent regions this may not be practical. The superelastic nature of the 60NiTi implies that when a stressed region is cut, the residual stresses will be relieved (a spring-back effect) and thus cross sections may not be truly representative. Additional investigations utilizing other approaches such as x-ray neutron diffraction and TEM of severely deformed thin sections may be required to understand the micro-structural phenomena controlling elastic response to pre-stressing. Such research is expected to yield better understanding of the

mechanisms at play and also may help guide alloy and processing development. For the present, it is sufficient to simply recognize that more fully elastic behavior results when 60NiTi surfaces are exposed to high stress levels prior to entering service in the highly loaded contacting portions of mechanical components.

The ability to increase the contact stress (or load) beyond which permanent deformation occurs creates an opportunity to enhance the resiliency of 60NiTi provided a means for pre-stressing could be developed. Based upon the results presented in this paper, several key aspects of a method for effective pre-stressing become apparent. Since the levels of stress required to yield an improvement in resiliency would cause excessive deformation in unhardened state, pre-stressing must be done to surfaces that have already been heat treated to attain high hardness. In addition, the heat treatment process occurs at a temperature that is above the solvus temperature for NiTi and any beneficial pre-compression effects observed in unhardened material would be relieved prior to the quenching step. Further, since the quenching step following solution treating results in the formation of significant residual stresses, major material removal on a 60NiTi component after hardening and pre-stressing must be minimized to avoid stress relation associated geometrical distortions. When these factors are taken into consideration, the pre-stressing of a precision component like a ball bearing appears to be a formidable task, albeit a task with significant potential benefits.

Future efforts will be aimed at developing techniques to effectively pre-stress the contacting surfaces of complex mechanical components such as ball bearings, gears and other aerospace mechanisms. The general method currently under consideration involves rough manufacturing of a component, like a bearing raceway, and applying forces and stresses similar to but at higher levels than those likely to be encountered in service. Such a technique will be the subject of follow on research papers.

### **Summary Remarks**

60NiTi is an attractive candidate alloy for aerospace bearing applications because it is hard and resilient. Static indentation load capacity tests show that 60NiTi surfaces can withstand higher loads than conventional bearings materials. 60NiTi surfaces that are additionally processed by applying high levels of compressive stress, exhibit a significant load capacity increase. For surfaces pre-stressed to 2.70 GPa, the indentation load capacity increases by about 30 percent. These results are consistent with the compressive behavior observed for 60NiTi measured in standard compression tests. Compared to the corrosion resistant steel, 440C, pre-stressed 60NiTi has three times the static load capacity. Compared to REX 20, the very strongest tool steel (that lacks corrosion resistance), pre-stressed 60NiTi has about twice the static load capacity.

Though the exact metallurgical mechanisms for the observed effects are not yet known, the results obtained are helpful in guiding an engineering method to enhance the resiliency of mechanical components like ball bearings. Further investigations in this vein are expected to result in new methods for producing corrosion proof, shockproof mechanical components made from superelastic materials that may alleviate many aerospace mechanism performance challenges.

## References

- 1. DellaCorte, C., Pepper, S.V., Noebe, R., Hull, D.R., and Glennon, G., 2009, "Intermetallic Nickel-Titanium Alloys for Oil-Lubricated Bearing Applications," NASA/TM—2009-215646.
- DellaCorte, C., Noebe, R., Stanford, M.K., and Padula, S.A., "Resilient and Corrosion-Proof Rolling Element Bearings Made From Superelastic Ni-Ti Alloys for Aerospace Mechanism Applications," Proceedings of the 2011 ASTM Rolling Element Bearings Conference, Anaheim, CA, April 13-15<sup>th</sup>, 2011 and NASA/TM—2011-217105.
- 3. Timoshenko, S.P., and Gere, J.M.: Mechanics of Materials, Chapter 1, pp. 3-10, published by Brooks/Cole, Monterey, CA, 1972.

- 4. W. Park, M.R. Hilton, P.C. Ward, G.W. Henderson, A. R. Leveille, D.E. McClintock, and D.W. Smith, "Microstructure, Fatigue Life and Load Capacity of PM Tool Steel REX20 for bearing Applications," Lubrication Engineering, volume 55, number 6, pp. 20-30, 1999.
- 5. A. Stebner, S.A. Padula, R.D. Noebe, and D.D. Quinn, "Characterization of Ni19.5Ti50.5Pd25Pt5 High Temperature Shape memory Alloy Springs and Their Potential Application in Aeronautics," Proceedings of SPIE Vol. 6928, 6928X, 2008.
- C. DellaCorte, Moore, L.E. III, and Clifton, J.S.: "Static Indentation Load Capacity of the Superelastic 60NiTi for Rolling Element Bearings", NASA/TM—2012-216016, presented at the 2012 STLE Annual Meeting, St. Louis, MO, May 6-10, 2012.
- 7. W.J. Buehler, Private communication with authors, Nov. 2008.
- W.J. Buehler, "Intermetallic Compound Based Materials for Structural Applications," Proceedings of the 7th Navy Science Symposium: Solution to Navy Problems Through Advanced Technology, May 14-16, 1963, U.S. Naval Aviation Medical Center, Pensacola, FL, Volume 1, Office of Naval Research, Arlington, VA, May 1963.
- 9. W.J. Buehler, J.V. Gilfrich, and R.C. Wiley, "Effect of Low-Temperature Phase Changes on the Mechanical Properties of Alloys near Composition TiNi," Journal of Applied Physics, vol. 34, no. 5, May 1963, pp. 1475–1477.