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Fransua Thomas
Glenn Research Center, Cleveland, Ohio

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The Effect of Various Quenchants on the Hardness and Microstructure of 60-NITINOL

Fransua Thomas
National Aeronautics and Space Administration
Glenn Research Center
Cleveland, Ohio 44135

Abstract

The effect of various quenching media on the hardness and microstructure of 60 NITINOL (60 NiTi) were evaluated. Specimens of 60 NiTi were heat treated in air at 1000 °C for 30 min or 2 hr, then quench cooled by one of seven different methods. The microstructure and hardness of this material was examined post heat treatment. The results indicated that the quench method had little effect on the resulting hardness and microstructure of 60 NiTi.

Introduction

There is an increasing interest in 60 NITINOL (60 NiTi) as an alternative material for bearing and gear applications (Ref. 1). In spacecraft systems, bearings must withstand transient loading from the vibration of launch and resist wear-accelerating corrosion from the operating environment as well as from the corrosive ambient marine environment of many of the U.S. spaceports. Consisting of 60 wt% Ni and 40 wt% Ti, 60 NiTi is characterized by its excellent hardness, wear resistance, dimensional stability, corrosion resistance and tribological properties (Ref. 2).

Heat treatment is an important process to attain maximum hardness in NiTi alloys like 60 NiTi. The process includes heating the material to high temperatures followed by rapid cooling. The heating step is referred to as *solution treating*. During this process the material is heated to a temperature above the solvus point and held there for sufficient time to permit the formation of a uniform NiTi phase. For, 60 NiTi this temperature is 900 to 1050 °C and the soak period is 30 to 120 min. The next step is quenching or rapid cooling. Quenching is then used to retain the generally preferable austenitic phase of the 60 NiTi. Unlike bearing steels in which the martensitic phase is desired due to its high hardness, in NiTi alloys it is the austenitic phase that is hard and sought after in bearing applications.

Rapid cooling during heat treatment can, however, produce internal residual stresses that can lead to dimensional distortion and, in extreme cases, quench cracking, where thermally-induced stresses exceed the tensile strength of the material (Ref. 3). Oftentimes, the heat treatment process must be adjusted to strike a balance between hardness and residual stress levels. The principle objective of this study is to understand the effect of the cooling rate of various quechants on the hardness and microstructure of 60 NiTi and to improve our understanding of the overall heat treatment process.

Materials and Procedures

The 60 NiTi (60 wt% Ni – 40 wt% Ti) was obtained from commercial sources. Specimens were cut from an ingot of the material made by hot isostatically pressing atomized Ni - Ti powder in a steel container. The specimens used for this heat treatment investigation were approximately 45.7×3×4 mm. The specimens were sectioned from the ingot by electrical-discharge machining (EDM), placed in different quenchant groups then heat treated in air using a tube furnace. Specimens were heat treated for either 30 min or 2 hr at 1000 °C then quenched with one of the seven different quenching media. The quenchants used in this study represent commonly used quenchants: room temperature water, boiling water, ice water, compressed air, vegetable oil, glycol, and liquid nitrogen (LN_2).

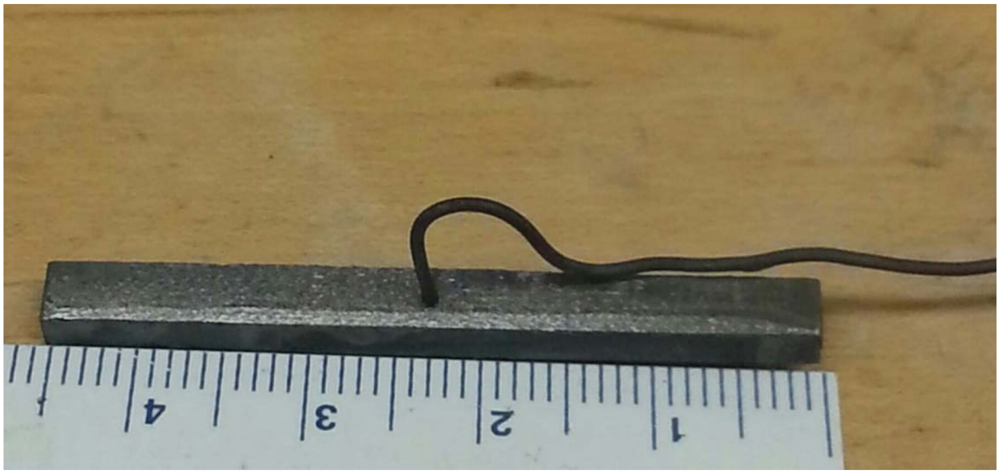

Figure 1.—Picture showing specimen and location of thermocouple.

Each specimen was fitted with a type K high temperature thermocouple, which was securely placed in the center of each specimen in a 1.50 mm bored hole (Fig. 1). The cooling curve data was recorded using a custom high speed acquisition program in which the millivolt signal from the embedded thermocouple was routed into the computer with an ICPDAS I-7019R data acquisition module. The program captured the data at a rate of approximately 30 Hz and exported it to a tab delimited text file.

After the solution treating step, the specimens were quickly removed from the furnace and subjected to the selected quenchant. To determine the hardness of the specimen, a standard Rockwell C indentation tester with a load of 150 kg was used. Prior to checking hardness, the thin oxide layer that formed during the heat treatment was removed by manual surface grinding. Four measurements were made on each specimen and the average of these measurements is reported. The metallographic process used to prepare the specimen consisted of a wet polished by hand using SiC abrasive paper starting with 320 grit abrasive and ending with 800 grit abrasive. Secondly a final polish used colloidal silica on a vibratory polisher which resulted in a near mirror. Lastly the polished surface was etched in a solution of 5 ml HF + 2.5 ml HNO_3 + 43 ml deionized water. The microstructure was observed by optical microscopy.

Results and Discussion

The average Rockwell hardness values obtained with each quenchant after heat soaks of either 30 min or 2 hr are shown in Tables 1 and 2, respectively. For the test conditions encompassed by this research, each quenchant yielded very similar results in hardness. The average hardness in each of the groups ranged from 59 to 61 HRC and the standard deviation was small, less than 1 hardness point. In other words, neither the solution treatment duration, nor the quenching media significantly affected the hardness for the variables considered in this study.

To obtain bearing quality hardness, specimens are rapidly cooled by quenching. This process is quite severe and may cause thermal cracking or distortion and this phenomena is the primary motivation for this study. The 60 NiTi specimens tested appear to be insensitive to the quenching media. The quenchants tested have varying thermal properties, leading to differences in the initial cooling rate. A comparison of the cooling curves obtained from seven different quenchants is shown in Figure 2.

To determine if the varying quenching media had any effect on the microstructure, metallographic characterization was conducted. Optical microscopy of cross sections of the specimens were carried out to compare the microstructures. The microstructure of the specimens after simple heat treatment that included solution heating at 1000 °C followed by rapid quench, shows second phase (Ni_3Ti) found throughout grains and grain boundaries. Figure 3 shows photomicrographs of the similar microstuctures of heat treated specimens quenched in the different quenchants. These similarities in microstructure and hardness are achieved through the rate in which the specimens are cooled in the fore-mentioned figure.

TABLE 1.—SUMMARY OF INDENTATION OF NITINOL SPECIMEN IN VARIOUS QUENCHANTS, SOLUTION TREATED FOR 30 MIN

Rockwell (C) hardness at 30 min	
Quenchant type	Average hardness (HRC)
RT water	61±0.5
Shop air	60±0.6
Boiling water	60±0.3
Ice bath	60±0.1
Oil	60±0.4
Glycol	60±0.2
LN_2	60±0.5

TABLE 2.—SUMMARY OF INDENTATION OF NITINOL SPECIMEN IN VARIOUS QUENCHANTS, SOLUTION TREATED FOR 2 HR

Rockwell (C) hardness at 2 hr	
Quenchant type	Average hardness (HRC)
RT water	60±0.2
Shop air	59±0.3
Boiling water	60±0.5
Ice bath	59±0.6
Oil	60±0.4
Glycol	60±0.1
LN_2	59±0.5

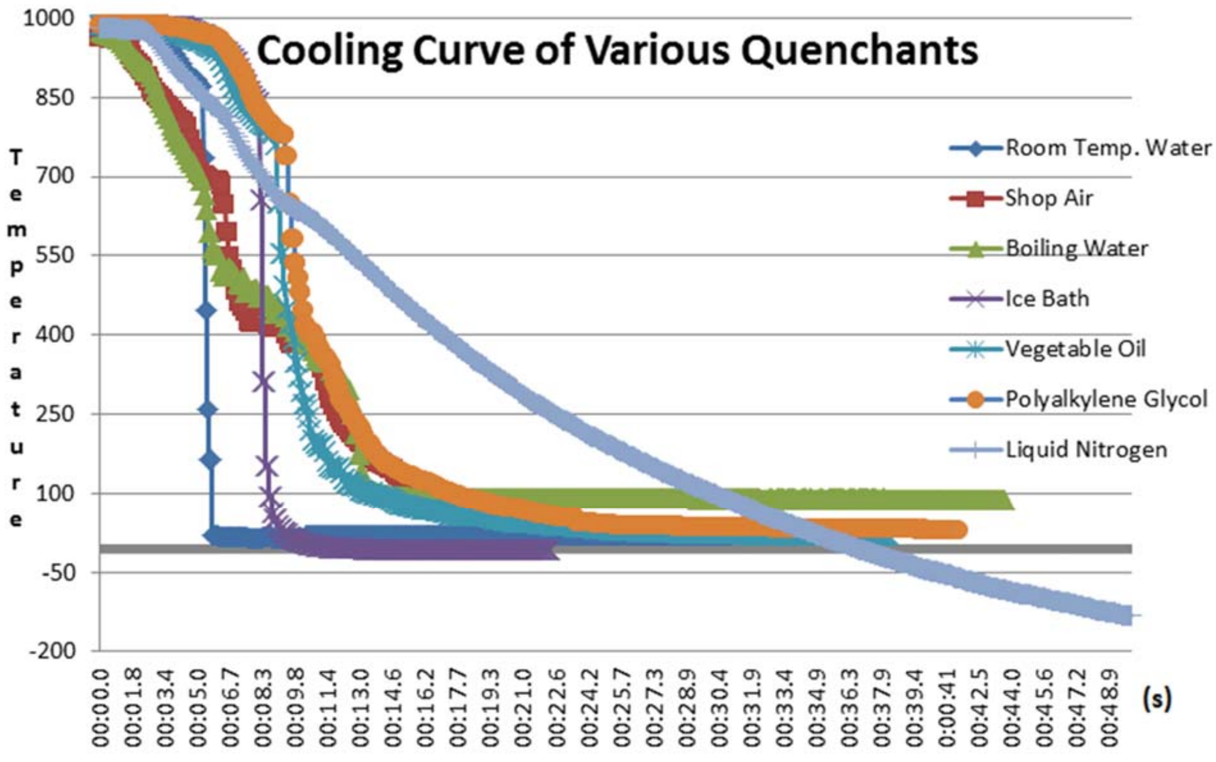

Figure 2.—Cooling curves for 60-NITINOL specimen in various quenchants.

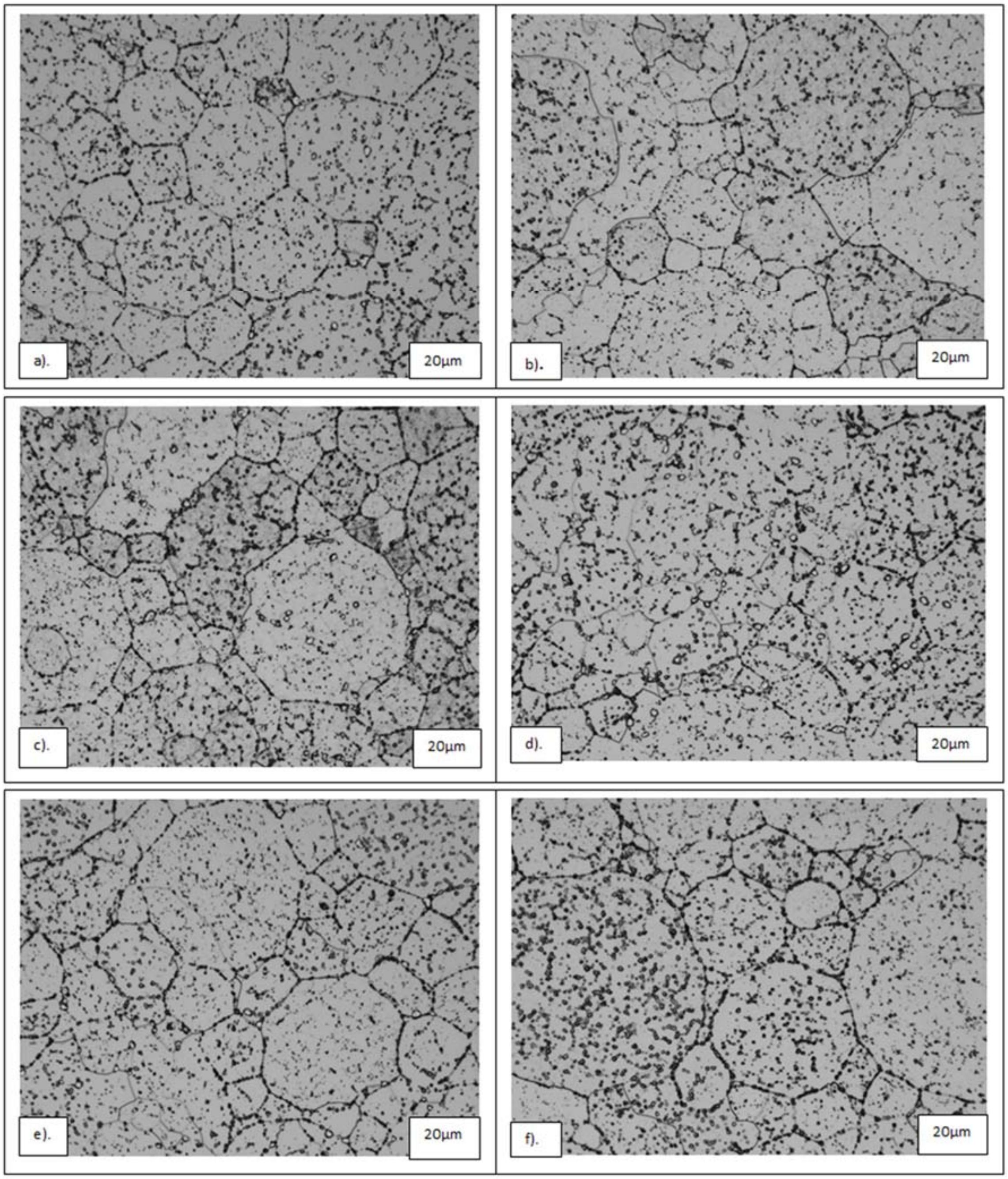

Figure 3.—Optical micrographs showing the microstructure of 60-NITINOL after heat treatment and various quenching mediums: (a) room temperature water, (b) boiling water, (c) ice water, (d) shop air, (e) vegetable oil, (f) glycol, and (g) liquid nitrogen (LN2).

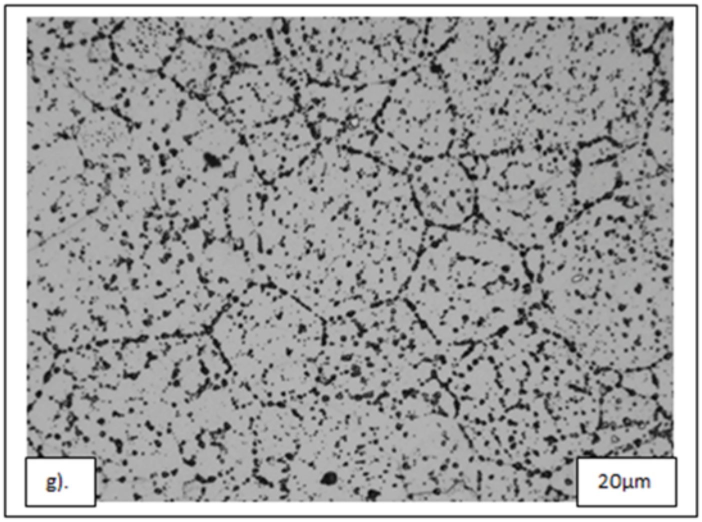

Figure 3.—Concluded.

Summary and Closing Remarks

The objective of this study is to understand the effect of various quenchants on the hardness and microstructure of 60 NiTi. Based on this study, the results show 60 NiTi exhibits high hardness (58 to 61 Rockwell C scale) when heat treated at 1000 °C for 30 min or 2 hr, regardless of which of the studied quenchants used for this specimen geometry. These results show that cooling rate is a factor in the hardness, when compared to earlier studies of larger specimens, suggesting that additional testing on larger specimens will have to be done to further this investigation.

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