



Cooling Rate Study of Nickel-Rich Material During Thermal Treatment and Quench

Fransua Thomas
Glenn Research Center, Cleveland, Ohio

Silvia Briseño Murguía
University of North Texas, Denton, Texas

NASA STI Program . . . in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program plays a key part in helping NASA maintain this important role.

The NASA STI Program operates under the auspices of the Agency Chief Information Officer. It collects, organizes, provides for archiving, and disseminates NASA's STI. The NASA STI Program provides access to the NASA Technical Report Server—Registered (NTRS Reg) and NASA Technical Report Server—Public (NTRS) thus providing one of the largest collections of aeronautical and space science STI in the world. Results are published in both non-NASA channels and by NASA in the NASA STI Report Series, which includes the following report types:

- **TECHNICAL PUBLICATION.** Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA counter-part of peer-reviewed formal professional papers, but has less stringent limitations on manuscript length and extent of graphic presentations.
- **TECHNICAL MEMORANDUM.** Scientific and technical findings that are preliminary or of specialized interest, e.g., “quick-release” reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.
- **CONTRACTOR REPORT.** Scientific and technical findings by NASA-sponsored contractors and grantees.
- **CONFERENCE PUBLICATION.** Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or co-sponsored by NASA.
- **SPECIAL PUBLICATION.** Scientific, technical, or historical information from NASA programs, projects, and missions, often concerned with subjects having substantial public interest.
- **TECHNICAL TRANSLATION.** English-language translations of foreign scientific and technical material pertinent to NASA's mission.

For more information about the NASA STI program, see the following:

- Access the NASA STI program home page at <http://www.sti.nasa.gov>
- E-mail your question to help@sti.nasa.gov
- Fax your question to the NASA STI Information Desk at 757-864-6500
- Telephone the NASA STI Information Desk at 757-864-9658
- Write to:
NASA STI Program
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681-2199



Cooling Rate Study of Nickel-Rich Material During Thermal Treatment and Quench

Fransua Thomas
Glenn Research Center, Cleveland, Ohio

Silvia Briseño Murguía
University of North Texas, Denton, Texas

National Aeronautics and
Space Administration

Glenn Research Center
Cleveland, Ohio 44135

This report contains preliminary findings,
subject to revision as analysis proceeds.

Trade names and trademarks are used in this report for identification
only. Their usage does not constitute an official endorsement,
either expressed or implied, by the National Aeronautics and
Space Administration.

This work was sponsored by the Fundamental Aeronautics Program
at the NASA Glenn Research Center.

Level of Review: This material has been technically reviewed by technical management.

Available from

NASA STI Program
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681-2199

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
703-605-6000

This report is available in electronic form at <http://www.sti.nasa.gov/> and <http://ntrs.nasa.gov/>

Cooling Rate Study of Nickel-Rich Material During Thermal Treatment and Quench

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

Silvia Briseño Murguía
University of North Texas
Denton, Texas 76203

Abstract

To investigate quench cracking that results from water quenching after heat treatment of binary and Ni-rich material, cooling rates of specimens were measured during quenching and hardness post-thermal treatment. For specific applications binary Ni-Ti is customarily thermally treated and quenched to attain desired mechanical properties and hardness. However, one problem emerging from this method is thermal cracking, either during the heat treatment process or during the specimen's application. This can result in material and equipment failure as well as financial losses. The objective of the study is to investigate the internal cooling rate of 60-NiTi during quenching and determine possible factors causing thermal cracking. Cubic (1 in.³) samples of both material were heat treated in air at 1000 °C for 2 hr and quenched in room temperature water using two methods: (1) dropped in the water and (2) agitated in the water. Hardness of the two fore-mentioned methods was measured post heat treatment. Results indicate that the quenching method had an effect on cooling rate during quenching but hardness was observed to be essentially the same through the thickness of the samples.

1.0 Introduction

The 60-Nitinol, composed of 60wt% Ni and 40wt% Ti, has been an increasingly attractive material in various industries due to its unique properties. The combination of low density, high strength, high hardness, low apparent elastic modulus and resistance to aqueous corrosion allows for a wide range of engineering applications such as bearing, gears, and other mechanical components (Ref. 1). To obtain the desirable high hardness, the Ni-rich material is heat treated. These thermal treatments include solution treating by heating and holding the alloy to above the solvus point until a uniform Ni-Ti phase is formed. The solution treating step is then immediately followed by a quench or rapid cooling which retains the generally preferable austenitic phase of Nitinol. However, the heat treatment process can produce internal stresses that can lead to distortion and in some cases these thermally induced stresses can exceed the tensile strength of the material and result in quench cracking (Ref. 2). A study by M. K. Stanford on the effects of high temperature heat treatments of 60-Nitinol showed that several ruptures of prototype components have occurred during the water-quenching step of the heat treatment process. The study also mentioned that cracking during quenching also occurred during heat treatment on relatively simple geometries, suggesting that residual stress within a component at stress concentration points such as fillets with high radii of curvature were not the only contributors to cracking (Ref. 3). The purpose of this report is to investigate the internal cooling rate of 60-Nitinol during thermal treatment and quenching, to determine the possible factors causing thermal cracking and to verify uniform cooling and hardening throughout the specimen.

2.0 Materials and Experimental Procedures

Two cubic specimens of 60-Nitinol, obtained from a commercial source, were cut from an ingot of material processed by hot isostatic pressing (HIP) atomized Ni-Ti powder in a steel container. The cubic specimens were cut by electrical-discharge machining (EDM) to approximately 1 in.³. Three 1/16 in. cavities of different depths were made along the center of the top face of the specimen for thermocouple placement. A type K, high temperature thermocouple was placed in each cavity and secured with wire (shown in Fig. 1). The thermocouples were placed at a depths of 0.25, 0.50, and 0.75 in. from the top surface. The thermocouples were linked to a computer which recorded temperature versus time using in-house software.

These specimens were then thermally treated in a tube furnace at 1000 °C for 2 hr in air and immediately quenched by one of two approaches. One method of quenching was done by placing the heated specimen into room temperature water and allowing it to reach the bottom of container and cool. This method will be referred to as immersion method. The second quench method is by agitation where the specimen was moved up and down in room temperature water until cool. This will be referred to as agitation method. Samples of the surface and center of these specimen were sectioned for hardness measurements. To determine the hardness of the specimen, the thin oxide layer that formed on the surface during thermal treatment was removed by surface grinding. The hardness measurements were taken using a standard Rockwell C indentation tester with a load of 150 Kg. Four hardness measurements were taken on each specimen and the average hardness was recorded.

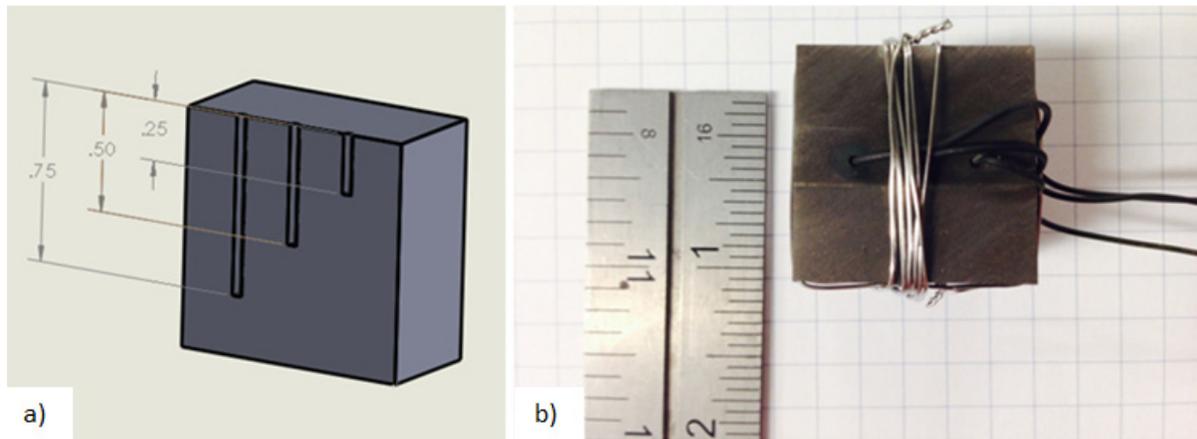


Figure 1.—(a) Schematic of specimen cross-section showing depth of the thermocouple holes in the specimen.
(b) Picture of Nitinol specimen and location of the thermocouples.

3.0 Results and Discussion

Specimens of both methods fractured during the quenching process. The temperature versus time comparison of the three depth locations and the two quenching approaches are shown below in Figures 2 and 3. The temperature curves of the specimen using the first quenching approach show a slight difference in cooling rate. However, the cooling trend is the same for all three curves indicating uniform cooling. The specimen's sudden drop in temperature seen at 30s in Figure 2 and 21s in Figure 3, denotes the instant the specimen fractured. A larger difference in the cooling of the specimen is observed in the agitation method. When comparing the two temperature graphs, the agitation method decreased the cooling time at the surface by 20 sec. The immersion method yielded steady-state temperature after approximately 50 sec, while the agitation method reached steady-state temperature at approximately 30 sec. The temperature curves in Figure 2 show greater variation between the cooling curves with respect to thermo-couple placement. The agitated specimen at 0.25 in. from the surface cooled the fastest, reaching 100 °C within 10 sec. Note, this is due in-part to several stages the specimen undergoes in-which

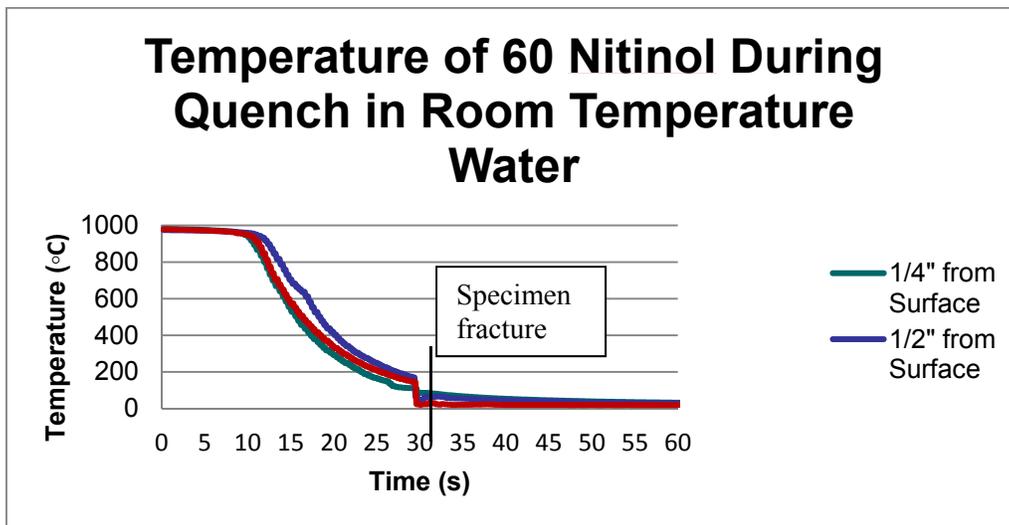


Figure 2.—Temperature curves for three different locations of a 60-NITINOL specimen during quench in water at room temperature.

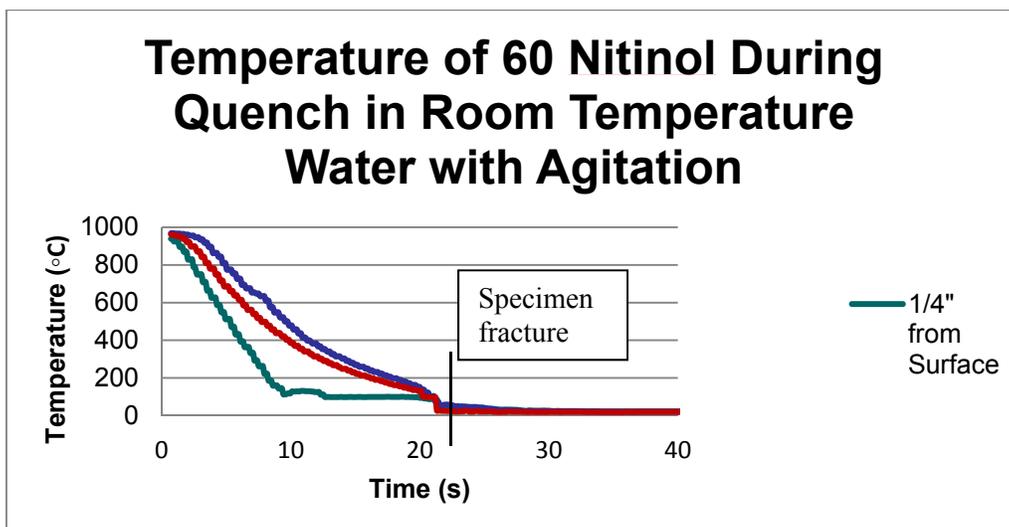


Figure 3.—Temperature curves for three different locations of a 60-NITINOL specimen during quench in water at room temperature with agitation.

agitation combats during quenching. First, a vapor blanket surrounds the specimen, temporarily insulating the specimen. Next, the specimen is engulfed with bubbles during the nucleate boiling stage providing high heat transfer rate. Lastly, during the convection stage, water is in direct contact with the specimen, allowing continued heat transfer (Ref. 4). This specimen thermally cracked, cracking at the 21 sec mark, indicated by a sharp drop in temperature (see Fig. 2).

The time differences between the time of fracture and final temperature suggest cooling rate is a factor in the formation of internal stresses within specimen.

Figure 4 shows the fractured remnants of the specimen that was agitated and during the quenching process. The fractured sample was preserved and had pieces large enough for hardness testing.

The average Rockwell hardness measurements from a section near the center of the specimens and at the surface are shown in Table 1. Hardness measurements from an annealed specimen are also shown below as a control.

The hardness of the immersion and agitation methods ranged from 59.2 to 60.5 HRC between the surface and center locations. The small difference in hardness between the surface and the center of the specimen suggests that there is consistent hardening throughout the specimen.



Figure 4.—(a) Fractured remains after thermal treatment using immersion method. (b) Fractured remains after thermal treatment using agitation method.

TABLE 1.—POST-THERMAL TREATMENT AVERAGE ROCKWELL C HARDNESS AT CENTER AND SURFACE OF THE IMMERSION SPECIMEN

Location of Nitinol specimen	Average Rockwell C hardness
Surface	59.8
Near the Center	59.4

TABLE 2.—POST-THERMAL TREATMENT AVERAGE ROCKWELL C HARDNESS AT CENTER AND SURFACE OF THE AGITATED SPECIMEN

Location of Nitinol specimen	Average Rockwell C hardness
Surface	60.5
Near the Center	59.2

TABLE 3.—POST-THERMAL TREATMENT AVERAGE ROCKWELL C HARDNESS AT CENTER AND SURFACE OF THE ANNEALED SPECIMEN

Location of Nitinol specimen	Average Rockwell C hardness
Surface	45
Near the Center	44.3

4.0 Conclusions

The objective of this investigation was to gain some understanding on the causes of thermal cracking in 60-Nitinol due to the thermal treatment process by investigating internal cooling rates during quenching. The results showed that the specimen exhibited only an average of 0.85 hardness difference between the center and its surface. With an average of hardness of 60.2 HRC at the surface and 59.3 HRC at the center of both specimen, this investigation exhibited consistent hardness throughout the specimen after the heat treatment process. The specimens did crack during the quenching process but the temperature versus time curves showed that quenching may have played a role in the observed quench cracking. By agitating the specimen in the quenchant rather than immersing the specimen in the quenchant, the cooling time is reduced. However, it is speculated that this faster cooling contributes to the internal stresses leading to quench cracking. Another possible factor leading to the fracture of each specimen are the holes used for thermocouple placement. Looking at the identical nature in which each specimen fractured the hole are indicative of crack initiation. These results suggest that additional testing such as chemical analysis of the material composition for contamination and high level magnification such as SEM for microstructural analysis will be needed to further investigate the thermal cracking contributors. Further work will include exploration of other heat treatment methods, geometries as well as other alloys such as ternary alloys like Ni-Ti-Hf.

References

1. M.K. Stanford, "Thermophysical properties of 60-NITINOL for Mechanical Component Applications," NASA/TM—2012-216056, December 2012, National Technical Information Services, Springfield, VA.
2. F. Thomas, "The Effect of Various Quenchants on the Hardness and Microstructure of 60-NITINOL," NASA/TM—2015-218463, January 2015, National Technical Information Services, Springfield, VA.
3. M.K. Stanford, "Processing Issues for Preliminary Melts of the Intermetallic Compound 60-NITINOL, NASA/TM—2012-216044, November 2012, National Technical Information Services, Springfield, VA.
4. Herring, Daniel H. "Oil Quenching," *Www.heat-treat-doctor.com.*, 2010. Web. 16 February 2016.

