Thermophysical Properties of 60-NITINOL for Mechanical Component Applications

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Abstract

The linear thermal expansion coefficient, specific heat capacity, electrical resistivity and thermal conductivity of 60-NITINOL were studied over a range of temperatures representing the operating environment of an oil-lubricated bearing. The behavior of this material appears to follow well-established theories applicable to either metal alloys, in general, or to intermetallic compounds, more specifically and the measured data were found to be comparable to those for conventional bearing alloys.

Introduction

60-NITINOL is an ordered intermetallic material composed of 60wt%Ni and 40wt%Ti. Due to its unique combination of high hardness, low apparent elastic modulus and resistance to aqueous corrosion, this material is of interest within the aerospace community for use as a bearing material (Refs. 1 and 2). 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. Unlike its extensively-studied cousin 66-NITINOL, however, the thermophysical properties of 60-NITINOL remain virtually unexplored. Knowledge of these properties will enable better design and modeling of components. The purpose of this investigation is to determine selected thermophysical properties of 60-NITINOL and compare them to those of conventional bearings to enable better design and modeling of components.

Materials

Gas atomized 60-NITINOL (60wt% Ni–40wt% Ti) powder was consolidated into an approximately 20 kg ingot by hot isostatic pressing. The microstructure of the hot isostatically pressed material is shown in Figure 1. The chemical composition of the material was analyzed by inductively-coupled plasma emission spectroscopy and the results are listed in Table I.

Slightly oversized specimen blanks for were sectioned from the ingot by wire electrical-discharge machining (wire EDM). The specimen blanks were heat treated by furnace-cooling (softening) and by water-quenching (hardening) heat treatments as described in Table II. Photomicrographs of the microstructures resulting from each heat treatment are shown in Figure 2. Ni-Ti particles, not consolidated during hot isostatic pressing, were observed throughout the material (see Figure 2). The effect of these unconsolidated particles, thought to be a result of oxidation of the Ni-Ti material during atomization, has not been studied extensively in relation to thermophysical properties. Although unconsolidated particles were found at the fracture initiation sites in previous impact testing of this material, the impact energy did not vary significantly from that of material without unconsolidated particles (Ref. 3). Although these features represent surface defects within the material, their effect on the studied thermophysical properties is currently unknown.

The crystalline phases present in the material after both heat treatments were identified by x-ray diffraction using Cu Kα radiation, as listed in Table III. Ni3Ti is a non-coherent phase that ostensibly does not contribute to hardening of the material while Ni4Ti3 is a fine phase that can be precipitated through aging to optimize hardness. Specimens for thermophysical property testing were sectioned from the heat treated blanks by wire EDM and then ground to final dimensions as listed in Table IV.

Test Methods

The coefficient of thermal expansion, specific heat capacity, thermal conductivity and electrical resistivity were measured for 60-NITINOL after furnace-cooling and water-quenching heat treatments. The temperature ranges were selected to include the upper limit of temperature experienced by oil-lubricated components but certain atmospheres were selected to eliminate the effect of oxidation on the property of interest.

Coefficient of Thermal Expansion

The linear coefficient of thermal expansion α is determined by measuring the relative change in length l with respect to temperature according to the relationship:

$$\alpha = (\Delta l/l)/(T - T_0).$$

where T is the test temperature and T₀ is room temperature. The test was performed in helium using a push-rod dilatometer.
Figure 1.—Scanning electron microscope photomicrograph of the compacted material showing clusters of second phase Ni$_3$Ti within the grains and along the grain boundaries of the parent NiTi phase.

| TABLE I.—CHEMICAL COMPOSITION OF COMPACTED 60-NITINOL POWDER |
| Composition | 59.4wt%Ni–40.5wt%Ti |
| Impurities (ppm) | O (580), Fe (100), Cu (90), Al (80), Cr (20), Co (5) |

| TABLE II.—HEAT TREATMENTS USED IN THIS STUDY |
| Designation | Heat treatment |
| Water-quenched | 2 h at 1000 °C then quenched in room temperature water (specimen agitated during immersion) |
| Furnace-cooled | 2 h at 1000 °C then allowed to cool inside closed furnace after it was powered-down |

| TABLE III.—CRYSTALLINE PHASE IDENTIFICATION |
| Water-quenched material | Furnace-cooled material |
| Cubic NiTi | Cubic NiTi |
| Hexagonal Ni$_3$Ti | Hexagonal Ni$_3$Ti |
| Rhombohedral Ni$_4$Ti$_3$ | Cubic NiTi$_2$ |
| Cubic NiTi$_2$ | Monoclinic NiTi |

| TABLE IV.—TEST SPECIMEN DIMENSIONS |
| Specimen type | Nominal dimensions |
| Thermal and electrical conductivity | 90- by 4.7- by 1-mm |
| Specific heat | 5.8-mm diameter by 1.5-mm |
| Thermal expansion | 50-mm diameter by 5-mm |

According to a standard test procedure (Ref. 4). The expansion of the specimen is compared to that of a platinum standard with a heating rate of 2 °C per minute. This validity of this technique as an evaluation tool assumes that the symmetry of the cubic B2 crystal structure will result in isotropic thermal expansion. For calibration of the instrument, six standards with a range of thermal expansion coefficients were obtained from the National Institute of Standards (NIST) and measured against one another.

Figure 2.—Photomicrographs showing 60-NITINOL in (a) water-quenched and (b) furnace-cooled heat treatment conditions (the green filter increases the resolution of the optical photomicrographs). Furnace-cooling allows precipitation of the second phase Ni$_3$Ti within the parent phase of the material.

**Specific Heat**

A differential scanning calorimeter was used to measure specific heat at constant pressure in argon according to a standard test specification (Ref. 5). The procedure measures the difference in power required to heat a 60-NITINOL specimen at the same rate as a sapphire standard. With the known masses of the specimen and the standard and the measured power differential, the specific heat $c_p$ is calculated as a function of temperature, following the well-known relationship

$$ c_p = q/m\Delta T $$

where $q$ represents the heat supplied to the material of mass $m$ to produce a change in temperature $\Delta T$. The measured values are directly traceable to NIST standards.
Thermal Conductivity and Electrical Resistivity

Thermal and electrical conductivity were measured in vacuum with a technique based on Kohlrausch’s Method (Refs. 6 and 7). In this test method, thermocouples, which serve as both temperature and voltage sensors, are affixed to the test specimen at both ends and at the center. The specimen is heated throughout its volume by Joule heating as a result of the application of direct current. The product of thermal conductivity $\kappa$ and electrical resistivity $\rho$ are determined by the relationship

$$\kappa \rho = \frac{(V_3 - V_1)^2}{4[2T_2 - (T_1 + T_3)]}$$

where $V_3 - V_1$ represents the voltage drop across the length of the specimen (the ends are arbitrarily labeled positions 1 and 3), $T_2$ is the temperature at the center (position 2) of the specimen and $T_1 + T_3$ represents the sum of the temperatures at both ends. The resistivity $\rho$ is simultaneously determined using the expression

$$\rho = \frac{(V_3 - V_1)}{AIL}$$

where $A$ is the cross-sectional area of the specimen, $I$ is the current and $L$ is the length of the specimen between positions 1 and 3. The current heats the specimen and radiant heat is supplied to the center of the specimen to minimize radial heat loss while the ends are cooled with a liquid medium. The current is varied to increase the resultant steady-state temperature profile as the test parameters are recorded. The measured values are directly compared to standards validated by the NATO Science and Technology Organisation (formerly the Advisory Group for Aerospace Research and Development) and are accurate to within 5 percent.

Results and Discussion

Coefficient of Thermal Expansion

A plot of the thermal expansion coefficient of 60-NITINOL from room temperature to 300 °C in both of the studied heat treatments is shown in Figure 3. The thermal expansion coefficient is slightly lower for the hardened material than for the softened material due to the increased presence of quenched-in defects, which restrict atomic mobility.

Specific Heat

The specific heat capacity is plotted in Figure 4. From 20 °C to approximately 300 °C, the heat capacity of the water-quenched material is slightly lower than that of the furnace-cooled material but the heat capacity for the water-quenched material increases at a slightly higher rate with respect to temperature. At 100 °C and above, the heat capacity of the water-quenched material is higher than that of the furnace-cooled material. The absence of any sharp deviations in the curve indicates that there are no phase transformations occurring over the reported temperature range.

Electrical Resistivity and Thermal Conductivity

Resistivity, plotted in Figure 5, shows the expected linear temperature dependence predicted by the Bloch-Gruenessien law (Ref. 8). It is noted that, while this behavior is not strictly followed at very low or very high temperatures, it is essentially linear for 60-NITINOL over the studied temperature interval. Electricity is conducted through metallic
materials by the positive drift of electrons or, more precisely, electron waves. Electrical resistivity tends to increase with temperature due to the concomitant increase in lattice vibration, which increases the probability of scattering these waves. As predicted by Matthiessen’s rule, irregularities such as point, line and surface defects also tend to increase resistivity due to scattering (Ref. 9). Water-quenching is known to create anti-phase boundaries that account for the increased resistivity in the water-quenched material (Refs. 10 and 11). Nevertheless, in both heat treatment conditions, the resistivity is in the range of typical metallic alloys (~10^{-7} \, \Omega \cdot m).

Simply comparing the measured resistivity values at room temperature and 300 °C in the two heat treatment conditions, the increase in resistivity resulting from changing the heat treatment condition from water-quenched to furnace-cooled is slightly less than the increase in resistivity resulting from increasing temperature over the range cited. This suggests that a non-destructive test could be developed to indicate the heat treatment condition of a component in the field exposed to unexpected current or heat transients as a possible “health monitoring” tool.

Thermal conductivity, plotted in Figure 6, is higher for the furnace-cooled material and increases with temperature in both heat treatment conditions. This behavior is similar to that of electrical conductivity. Unlike electrical current, heat can be conducted through a material by either electrons or phonons, a phenomenon expressed by the Wiedemann-Franz law (Ref. 12). For electrically conductive materials, the abundance of conducting electrons makes electronic conduction of heat more likely, which has been observed in other B2-type materials (Ref. 13). Based on the similarity of the electrical and thermal conductivity behavior in this material, it is likely that heat is primarily conducted by electrons in this material as well. Also, in contrast to the resistivity results, the percent increase in conductivity due to the difference in heat treatment condition appears to be essentially the same as the increase due to increasing temperature.

For ease of comparison, the average values of each of the measured properties are listed in Table V along with comparable values for several conventional bearing materials. It should be noted that, while the tabulated data from the literature is often reported as a single (averaged) value, it typically varies over the reported temperature range. Therefore, where warranted, a range of values is reported for 60-NITINOL.

The thermal expansion coefficient for 60-NITINOL is, perhaps unsurprisingly, intermediate to that of Ni and Ti and falls between that of 440C stainless steel and M50 tool steel. One might expect, therefore, that there would be little issue with thermal expansion mismatch if 60-NITINOL were mated with a steel component over the studied temperature range.

The range of reported values for the specific heat capacity of 60-NITINOL is comparable to that of M50 tool steel, 440C and 304 stainless steels and is only slightly higher than that of Stellite 6B and Hastelloy C. This is an indication that similar furnace energy usage requirements could be expected to heat treat 60-NITINOL as are found with steel heat treatment furnaces, given the same heat treatment temperature. This further indicates that it may be possible to use less aggressive quenchants (e.g., oils or polymers) to achieve a sufficient cooling rate for hardening heat treatments.
TABLE V.—COMPARISON OF THERMOPHYSICAL PROPERTIES FOR SELECTED MATERIALS AT ROOM TEMPERATURE (RT) EXCEPT WHERE SPECIFIED

<table>
<thead>
<tr>
<th>Material</th>
<th>Thermal expansion coefficient, $10^{-6}/^\circ$C</th>
<th>Specific heat capacity, J/kg·K</th>
<th>Thermal conductivity, W/m·K</th>
<th>Electrical resistivity, $\Omega$·m</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cobalt</td>
<td>13.1 (RT-200 °C)</td>
<td>414 (RT-100 °C)</td>
<td>69 (RT-100 °C)</td>
<td>$6.2 \times 10^{-8}$</td>
<td>14,15</td>
</tr>
<tr>
<td>Nickel</td>
<td>13.9 (RT-200 °C)</td>
<td>440 (RT-100 °C)</td>
<td>92 (RT-100 °C)</td>
<td>$8.0 \times 10^{-8}$</td>
<td>14,16</td>
</tr>
<tr>
<td>Titanium</td>
<td>9.5 (RT-300 °C)</td>
<td>519 (RT-100 °C)</td>
<td>22 (RT-100 °C)</td>
<td>$4.2 \times 10^{-7}$</td>
<td>14,16</td>
</tr>
<tr>
<td>304 stainless steel</td>
<td>17 (RT-300 °C)</td>
<td>500 (0-100 °C)</td>
<td>15.1 (RT-100 °C)</td>
<td>$7.2 \times 10^{-7}$</td>
<td>14,16</td>
</tr>
<tr>
<td>Stellite 6B</td>
<td>13.4 (RT-400 °C)</td>
<td>421</td>
<td>14.7</td>
<td>$9.1 \times 10^{-7}$</td>
<td>14,17,18,19</td>
</tr>
<tr>
<td>Hastelloy C</td>
<td>11.3 (0-100 °C)</td>
<td>419 (RT-100 °C)</td>
<td>11.5</td>
<td>$1.3 \times 10^{-8}$</td>
<td>14,20</td>
</tr>
<tr>
<td>M50 bearing steel</td>
<td>12.1 (RT-260 °C)</td>
<td>462 (RT-100 °C)</td>
<td>25.9 (RT-100 °C)</td>
<td>$1.8 \times 10^{-7}$</td>
<td>14,21,22</td>
</tr>
<tr>
<td>60-NITINOL (water-quenched)</td>
<td>11.2 (RT-300 °C)</td>
<td>453-504 (RT-300 °C)</td>
<td>8.9-13.7 (50-300 °C)</td>
<td>$1.0 \times 10^{-6}$ (50-300 °C)</td>
<td>23,24</td>
</tr>
<tr>
<td>440C stainless steel</td>
<td>10.8 (0-315 °C)</td>
<td>460 (0-100 °C)</td>
<td>41.9 (RT-150 °C)</td>
<td>$6.0 \times 10^{-7}$</td>
<td>14,16</td>
</tr>
<tr>
<td>Silicon nitride</td>
<td>3.0-3.5 (RT-1000 °C)</td>
<td>778 (127 °C)</td>
<td>22</td>
<td>$10^{10}$ (RT-100 °C)</td>
<td>25,26</td>
</tr>
</tbody>
</table>

The resistivity of 60-NITINOL is intermediate to that of Ni and Ti, being slightly higher than that of Stellite 6B and slightly lower than that of Hastelloy C. The thermal conductivity of 60-NITINOL is comparable to that of Hastelloy C and its upper limit is only slightly lower than the value given for Stellite 6B. These data indicate that 60-NITINOL would have the ability to dissipate electrical and thermal transients in service or during machining at least as well as the materials mentioned.

It is important to reiterate that, while “handbook” data such as those listed in Table V can provide a general overview of material properties, these properties typically vary with respect to the service temperature envelope and the service environment. Therefore, component designers should give these issues requisite consideration.

Conclusions

Selected thermophysical properties of 60-NITINOL have been studied and, based on this investigation, the following can be said:

- The thermal expansion coefficient, specific heat capacity, electrical resistivity and thermal conductivity of 60-NITINOL is comparable to a variety of conventional engineering alloys used in bearings.
- There is a significant difference in the thermophysical properties of 60-NITINOL in the water-quenched and furnace-cooled heat treatment conditions due to quenched-in defects.

References


