Low Temperature Mechanical Behavior of Ni-15Cr-Al-Ti-Mo Alloys

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A series of Ni-15 Cr-Al-Ti-Mo alloys with varying \(\gamma-\gamma'\) mismatch, Ti/Al atomic ratio, and weight fraction of \(\gamma'\) were tension tested at ambient temperature after aging to peak yield strength at 760°C. After subtracting the alloy yield stress in the solution treated condition, \(\sigma_{ys}\), the increment in yield stress due to precipitation of \(\gamma'\), \(\Delta\sigma_Y\), was found to be principally influenced by the weight fraction of \(\gamma'\) and the measured \(\gamma-\gamma'\) mismatch. Only a small contribution of APBE to alloy strength was observed between two compositions of similar mismatch and differing measured APBE. The work hardening behavior of the alloys was similarly influenced by weight fraction of \(\gamma'\) and \(\gamma-\gamma'\) mismatch. Alloys with low mismatch exhibited sheared \(\gamma'\) precipitates following tensile deformation. A model for shear of ordered \(\gamma'\) precipitates by residual dislocation loops in low \(\gamma-\gamma'\) mismatch alloys is proposed to account for the low work hardening rates observed.

Many commercial nickel-base alloys derive their superior mechanical properties from the coherent precipitation of \(\gamma'\), the intermetallic compound Ni\(_3\) (Al, Ti). The strengthening mechanisms which appear to contribute significantly to precipitation hardening in these \(\gamma-\gamma'\) alloys are order strengthening resulting from the \(\text{L}_{12}\) structure of the \(\gamma'\) precipitates and coherency strengthening derived from lattice mismatch between the \(\gamma\) and \(\gamma'\) phases. The relative importance of each mechanism is controversial, and investigators have reached differing conclusions regarding the role of coherency strains and antiphase boundary energy (APBE) in \(\gamma-\gamma'\) alloys with non-zero \(\gamma-\gamma'\) lattice mismatch.

The early studies by Nordheim and Grant\(^1\) showed an appreciable gain in the hardness and stress-rupture properties of Ni-Cr-Al alloys when Ti additions were made. Ti was found to substitute for Al in the precipitated \(\gamma'\), increasing its mismatch with the \(\gamma\) matrix phase. Mihalisin and Decker\(^2\) studied the room temperature and hot hardness of a series of Ni-Al-Ti binary and ternary alloys. A direct correspondence between age hardening response and lattice disregistry was observed for alloys in which the Ti/Al atomic ratio was varied. Mihalisin and Decker noted that the lattices of \(\gamma\) and \(\gamma'\) are both fcc and closely matched to allow coherency, and they concluded that the magnitude of age hardening in Ni-Al-Ti alloys was a function of coherency strains. In a subsequent study\(^3\) Decker and Mihalisin again demonstrated the importance of coherency strains in strengthening \(\gamma-\gamma'\) alloys. Selective ternary additions were made to a Ni-Al alloy to vary \(\gamma-\gamma'\) mismatch while monitoring the contributions of solid solution strengthening and APBE to age hardening.

Conflicting conclusions regarding the role of coherency strengthening in \(\gamma-\gamma'\) alloys have been reached by several authors. Phillips\(^4\) studied the reversible contribution to the flow stress of a Ni-Al alloy over a temperature range of 77 to 303 K. Analyzing the contributing factors to the alloy flow stress, Phillips concluded that coherency strains could account for only a small fraction of alloy strengthening while modulus mismatch and order strengthening were suggested as major contributors. Raynor and Silcock\(^5\) found little correspondence between \(\gamma-\gamma'\) mismatch and the increase in flow stress due to \(\gamma'\) precipitation in a series of \(\gamma'\) strengthened austenitic steels. Dislocation measurements indicated that Ti additions to these alloys increase the APBE of \(\gamma'\). Raynor and Silcock concluded that the strengthening of \(\gamma'\) containing alloys with low \(\gamma-\gamma'\) mismatch is attributable to the APBE of \(\gamma'\) and that the APBE varies with Ti/Al ratio. Most recently, studies on Ni-Al single crystals indicate that the contribution of coherency strains to alloy strengthening may depend on the volume fraction of \(\gamma'\) precipitate. Munjal and Ardell\(^6\) determined that the contribution of coherency hardening was negligible in a Ni-12.19 at. pct alloy with a volume fraction of \(\gamma'\) of 0.056. A subsequent study of alloys with large volume fractions of \(\gamma'\) inferred that a coherency strengthening mechanism was operative on the basis of a systematic temperature dependence of the flow stress.

The present study was initiated to determine the relative importance of ordering and coherency strains as particle strengthening mechanisms in \(\gamma'\) precipitating nickel-based alloys. The mechanical behavior of an alloy series with carefully controlled compositions was studied, within which alloy additions were used to provide wide ranges of \(\gamma-\gamma'\) mismatch, Ti/Al ratio, and fraction of \(\gamma'\). The microstructures of deformed specimens were observed in transmission electron microscopy, and the mechanical properties variations were related to \(\gamma'\) particle dislocation interactions.

MATERIALS

Three groupings of compositions within the Ni-Cr-Mo-Al-Ti system were chosen to study the respective effects of \(\gamma-\gamma'\) lattice mismatch, fraction of \(\gamma'\), and...
Ti/Al ratio. Table I lists the nominal compositions within these groupings and the major influence of alloy additions for each grouping. Alloy 14 was used as a control to determine the extent of solid solution strengthening. Since this alloy was not precipitation hardenable, it provided an estimate of the mechanical properties of the alloys in the absence of γ'. Small additions of carbon and boron were made to stabilize grain size and improved the hot working characteristics of these alloys.

The first grouping of alloys, 1 through 7, was designed to vary the magnitude of the unconstrained γ-γ' mismatch,

\[ \delta = \frac{a_\gamma' - a_\gamma}{a_\gamma} \text{ where:} \\
\]

\[ a_\gamma = \text{lattice constant of } \gamma, \text{ measured when separated from the bulk alloy, and} \\
\]

\[ a_\gamma' = \text{lattice constant of the matrix phase.} \]

The Ti/Al atomic ratio of alloys 1 through 4 was 1.8, chosen to yield a large initial mismatch between phases and to restrict the solubility of Mo in γ'. Mo additions of 0 to 5 at. pct were made to decrease 6 by increasing the γ lattice parameter and do not significantly alter the Ti/Al ratio of the γ' precipitate. Alloys 5 through 7 were designed to have a lower initial mismatch, δ, and to reduce 6 from positive to negative values as Mo was added. Ti, which lowers the solubility of Mo in γ, was not present in these alloys.

The second grouping, comprised of alloys 1, 5, 8 and 9, was designed to alter both the APBE and the lattice mismatch. For these alloys the Ti/Al atomic ratio varied from 0 to 5.4 with a constant (Ti + Al) content. Increases in both the γ' lattice constant and the APBE of γ'5 have been attributed to the substitution of Ti atoms for Al in the γ' lattice.

A third grouping, alloys 10 through 13, was designed to vary the fraction of γ' precipitate by increasing the total (Ti + Al) content. The Ti/Al atomic ratio was held constant at 1.8 to maintain a constant APBE and γ-γ' mismatch. Alloy 14 was used as a control to determine the extent of solid solution strengthening. This alloy was not precipitation hardenable and it provided an estimate of the mechanical properties of alloys in the absence of γ'.

Table I lists the actual compositions of the experimental alloy series in atomic percent. Alloys 1 through 9, and 14, were prepared by the Special Metals Corporation, New Hartford, N.Y. Starting materials were high purity elemental materials with the exception of boron, which was added as NiB. Seventeen pound heats were melted using the vacuum induction technique and cast to 9 cm. diam by 20 cm. long ingots. The hot top was removed and the ingots extruded at 1180°C to 2 cm. diam rod. Extrusion was performed by International Nickel Company, Inc., Sterling Forest, N.Y.

Alloys 10 through 13 were prepared by the Allegheny Ludlum Steel Corporation, Brackenridge, Pa. Starting materials were electrolytic grade elemental materials, with the exception of boron, added as NiB. Fifty pound heats were vacuum induction melted and cast into 10 cm. square molds. Ingots were hot rolled from 1150°C to produce a uniform cross section 9 cm. square, and rolled to 10 cm. x 90 cm. x 1 cm. plate above 1000°C.

**EXPERIMENTAL PROCEDURE**

The experimental procedure consisted of two parts: characterization and mechanical properties measurement. The experimental alloys were characterized in terms of microstructure, γ-γ' lattice mismatch, and weight fraction of γ'. Antiphase boundary energies were determined for alloys 4 and 5. Following characterization, the peak aged condition of each alloy was defined by hardness measurement after isothermal aging, and the tensile properties and deformation structures of peak aged specimens were studied.

**ALLOY CHARACTERIZATION**

Optical metallographic techniques were used to measure grain sizes and to prepare specimens for surface replication. Grain sizes of alloys solutionized at 1150°C for two hours and isothermally aged 480 h at 760°C were measured by counting the number of grains per mm². All of the alloys were coarse grained in this condition, with grain diameters decreasing from 0.2 mm to 0.1 mm with increasing Mo content. Chromium shadowed carbon replicas were prepared from these specimens and examined in transmission electron microscopy. The size, morphology, and distribution of the phases present at shorter aging times were determined on thin foil specimens using transmission electron microscopy.

Precision lattice parameter measurements were made to determine the values of \( a_\gamma' \) and \( a_\gamma \). An x-ray diffractometer technique employing a silicon standard minimized systematic errors, and provided a reference between specimens. Values of 2θ for the alloy 220 reflection of Cr Ka radiation were used to calculate \( a_\gamma \). All lattice parameter measurements were made on specimens aged 1000 h at 760°C. The \( a_\gamma' \) values were determined on bulk specimens. Powder samples of γ' obtained by electrolytic phase extraction from bulk samples were used to measure \( a_\gamma' \).

The compositional variation of the alloy series required that the fraction of γ' contained in each alloy be...