Grain Growth and Carbidic Precipitation in Superalloy, UDIMET 520

S. XU, J.I. DICKSON, and A.K. KOUL

The results of an experimental study on the grain coarsening behavior, M$_2$C$_6$ carbide precipitation, and secondary MC carbide precipitation kinetics in UDIMET 520 are presented. Primary MC carbides and M(C, N) carbonitrides strongly influence the grain growth, with their dissolution near 1190 °C and 1250 °C, respectively, resulting in two distinct grain coarsening temperatures (GCTs). M$_2$C$_6$ carbides precipitate in the alloy over a wide range of temperatures varying between 600 °C and 1050 °C. A discrete M$_2$C$_6$ grain boundary carbide morphology is observed at aging temperatures below 850 °C. Secondary MC carbides form at temperatures ranging between 1100 °C and 1177 °C, in specimens in which primary MC dissolution had been obtained at solution treatment temperatures of 1190 °C to 1250 °C. A schematic time-temperature-transformation (TTT) diagram for understanding the microstructure and precipitation inter-relationships in UDIMET 520 alloy is also presented.

I. INTRODUCTION

The properties of a material are determined by its chemical composition and microstructure. In superalloys, besides the size and distribution of the hardening intragranular precipitates and the dislocation substructure, the controlling microstructural features include the size and distribution of grains, the size, morphology, distribution, and nature of grain boundary precipitates, and whether the grain boundary morphology is planar or serrated. The grain size plays an important role in controlling the mechanical properties of superalloys, including the creep rupture[1] and the creep crack growth properties.[2] The influence of grain boundary precipitates (for example, M$_2$C$_6$ carbides) on the properties of Ni-base superalloys has also been recognized.[3-4] The kinetics of grain boundary precipitation and the precipitate distribution are critical. In order to optimize the creep rupture properties, a discrete distribution of grain boundary M$_2$C$_6$ carbides is usually preferred in superalloys after standard heat treatments. The importance of these microstructural features has been discussed at length in a recent comprehensive review article.[3] In addition to grain boundary M$_2$C$_6$ carbides, the MC carbide precipitation reactions also influence the properties of superalloys.[5-7] The primary MC carbides form during solidification. The secondary MC carbides may precipitate during an annealing or aging treatment at temperatures below the MC carbide solvus temperature, after cooling from a solution annealing temperature above this solvus temperature. The precipitation kinetics of both M$_2$C$_6$ and MC phases are mainly influenced by the chemical composition and the microstructural state prior to aging. The microstructural state prior to aging is governed by the solution treatment conditions employed and the relative magnitude of the solutionizing temperature with respect to the grain coarsening temperature (GCT). The GCT of an alloy is defined as the transition temperature above which grain growth occurs very rapidly within practical times.[5] The GCT of an alloy containing a high γ' volume fraction is often related to the solvus temperature of either γ phase or MC carbides.[5] Growth in low γ' volume fraction forged alloys usually begins when the solution temperature is sufficiently high to dissolve the primary carbides in the grain boundary regions. As the temperature surpasses the MC carbide solvus temperature, many of the primary MC carbides dissolve, and this is often accompanied by rapid grain coarsening. Other factors, such as prior deformation, prior particle boundaries in powder metallurgy (P/M) alloys, and the interdendritic eutectic γ-γ' pools in high γ' volume fraction cast alloys may also influence the grain coarsening behavior.[5-9] These observations indicate that the different pre- and postaging microstructural features in superalloys are inter-related and should be studied simultaneously. However, few studies have focused on these inter-relationships in a comprehensive manner.

UDIMET* 520 is a low volume fraction γ Ni-base superalloy, which is mainly employed for gas turbine parts, sheets, and bolts for industrial and marine use. The conventional heat treatment for wrought UDIMET 520 alloy involves a three-step treatment consisting of 1121 °C/4 h/AC, 843 °C/24 h/AC, and 760 °C/16 h/AC (where each step is given by the temperature, the time at this temperature, and the nature of the subsequent cooling, with AC indicating air cooling).[10,11] In some applications, a fourth stress relief step of 8 hours at 816 °C followed by air cooling is also employed.[12] After the conventional heat treatment,[10,11] UDIMET 520 microstructure is composed of a Ni-Cr-Co (γ) rich matrix, which is hardened by fine (approximately 0.1-μm size) Ni$_3$ (Al, Ti) gamma prime (γ') precipitates. Grain boundaries are decorated by M$_2$C$_6$ carbides.[13] Stringer-like primary MC carbides may also be present within the grains. Little information has been published on the grain growth and grain boundary carbide pre-

---

*UDIMET is a trademark of Special Metals Inc., New Hartford, NY.

S. XU, formerly Ph.D. Student at Ecole Polytechnique de Montreal, is with the Materials Technology Laboratory, CANMET, Natural Resources Canada, Ottawa, ON, Canada K1A 0G1. J.I. DICKSON, Professor, is with the Department of Metallurgy and Materials Engineering, Ecole Polytechnique de Montreal, Montreal, PQ, Canada H3C 3A7. A.K. KOUL, Senior Research Officer, is with the Structures, Materials and Propulsion Laboratory, Institute for Aerospace Research, National Research Council of Canada, Ottawa, ON, Canada K1A 0R6. Manuscript submitted April 28, 1998.
Table I. Chemical Composition of the Alloy Investigated (Weight Percent)

<table>
<thead>
<tr>
<th>Ni</th>
<th>Cr</th>
<th>Co</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>W</th>
<th>C</th>
<th>B</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bal</td>
<td>18.9</td>
<td>11.9</td>
<td>6.00</td>
<td>3.21</td>
<td>2.22</td>
<td>1.05</td>
<td>0.06</td>
<td>0.006</td>
<td>0.002</td>
</tr>
</tbody>
</table>

It is the purpose of this article to quantitatively study the grain growth and carbide precipitation behavior in UDIMET 520. Specifically, grain boundary M$_{23}$C$_6$ carbide precipitation kinetics and related microstructural changes, including the secondary MC carbide precipitation behavior, are studied in detail. A schematic diagram of the microstructural corelationships in UDIMET 520 is also developed.

II. EXPERIMENTAL PROCEDURE

A. Experimental Material

Wrought UDIMET 520 (U-520) nickel-base superalloy was employed in this study. The experimental material had been hot-rolled and centerline ground in the form of re-forging stock by Kelsey Hayes Inc. (Utica, NY). Table I lists the chemical composition of the material investigated.

B. Experimental Methods

1. Grain coarsening behavior

To study the effect of solution treatment conditions on the grain size, specimens were solution treated at 1050 °C, 1090 °C, 1135 °C, 1185 °C, and 1235 °C for 1, 2, and 4 hours followed by AC as well as at 1190 °C, 1200 °C, 1210 °C, and 1250 °C for 2 hours followed by water quenching (WC). A solution treatment of 2 hours was employed at different solution temperatures for establishing the grain growth curve, and the Vickers hardness of these solution-treated specimens was also measured. An immersion etchant containing 10 mL HNO$_3$, 10 mL acetic acid, 15 mL HCl, and 2 to 5 drops glycerol was employed to reveal the grain structure in solutionized specimens. The grain size was measured as the mean intercept grain length by the Heyn intercept method, with sufficient grains measured so that the results were within the 95 pct confidence limit in all cases. All Vickers microhardnesses reported correspond to an average of five measurements.

2. Carbide precipitation kinetics

The M$_{23}$C$_6$ grain boundary carbide precipitation kinetics study was conducted on two separate batches of specimens subjected to two different solution treatment conditions. For one batch, the solution treatment was carried out at 1135 °C for 4 hours followed by AC, and for the other, a solution treatment of 1250 °C for 2 hours followed by WC was employed. As expected, these solution treatment conditions produced significant differences in grain sizes. The aging experiments were conducted on cube-shaped specimens, 5 × 5 × 5 mm, in a radiation-type electric furnace. All specimens were polished and immersion etched following standard metallographic procedures. The etchant employed was three parts glycerol, two to three parts HCl, and one part HNO$_3$. At room temperature, M$_{23}$C$_6$ grain boundary carbides, if present, were revealed after etching for 60 to 90 seconds. This rapid method of verifying the presence or absence of grain boundary M$_{23}$C$_6$ carbides was initially calibrated against transmission electron microscopy (TEM) replica observations in conjunction with energy dispersive X-ray (EDX) microanalysis and scanning electron microscopy (SEM) observations. After etching for 90 to 120 seconds, the γ' was also easily revealed. First stage carbon replicas from the metallographic specimens were examined in a JEM-2000FX transmission electron microscope. Carbides were identified by SEM or TEM using EDX analysis and by TEM using the electron diffraction method. Vickers microhardness measurements at a load of 0.98 N or 100 gram-force were also performed on metallographically polished surfaces, which had been cut slowly with a diamond-coated saw blade. All Vickers microhardness numbers given represent an average of at least five measurements.

Specimens solution treated at a higher temperature of 1250 °C for 2 hours were employed to establish the secondary MC carbide precipitation temperature range. Metallography and SEM observations along with EDX microanalyses permitted identification of the secondary MC carbides. Primary MC carbides and M(C, N) carbonitrides were also identified by metallography and SEM observations in combination with EDX microanalysis employing a light element detector.

III. RESULTS AND DISCUSSION

A. Grain Growth in UDIMET 520

Figure 1 shows the microstructure of UDIMET 520 alloy forged stock in the as-received condition, in which the grain boundaries are somewhat difficult to etch. A necklace-type microstructure, consisting of small grains approximately 12 μm in diameter between larger grains approximately 82 μm in diameter, was present. Primary MC carbides, identified by EDX microanalyses, appeared as quite large (4 to 10 μm in width) blocky gray particles. Some carbonitrides, identified by EDX with light element detector, were also present and had an approximately cubic morphology.