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THESIS

THE EFFECTS OF THERMOMECHANICAL PROCESSING  
PARAMETERS ON ELEVATED-TEMPERATURE  
BEHAVIOR OF A 6061 Al-Al<sub>2</sub>O<sub>3</sub>  
METAL MATRIX COMPOSITE

by

Thomas J. Schauder

March, 1992

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| <p>THE EFFECTS OF THERMOMECHANICAL PROCESSING PARAMETERS ON THE ELEVATED TEMPERATURE BEHAVIOR OF A 6061 AL-AL2O3 METAL-MATRIX COMPOSITE (MMC) HAVE BEEN STUDIED. THE SAME PROCESSING PARAMETERS WERE EMPLOYED WITH UNREINFORCED 6061 AL TO PROVIDE A COMPARISON. THESE MATERIALS WERE BOTH THERMOMECHANICALLY PROCESSED AT EITHER 350 C OR 500 C USING TWO ROLLING SCHEDULES. BOTH SCHEDULES INVOLVED A CONSTANT STRAIN PER PASS. SUBSEQUENT MECHANICAL TESTS WERE CONDUCTED AT TEMPERATURES 200 TO 500 C AND STRAIN RATES RANGING FROM 6.7E-3 TO 1.3E-1 S-1. THE MATERIALS PROCESSED AT 500 C EXHIBITED HIGHER STRENGTH WHEN COMPARED TO THOSE PROCESSED AT 350 C FOR DEFORMATION TEMPERATURES BELOW 350 C. MATERIALS STABILIZED BY ANNEALING AFTER COMPLETION OF ROLLING DISPLAYED HIGHER DUCTILITIES WHEN COMPARED TO THE AS-PROCESSED MATERIALS. ESPECIALLY AT LOWER TESTING TEMPERATURES. THE PEAK DUCTILITIES OF THE MMCS OCCURRED AT TESTING TEMPERATURES NEAR THE PRIOR ROLLING TEMPERATURES. SOLUTION TREATMENT PRIOR TO ROLLING AND ADDITIONAL STRAINS DURING ROLLING IN EXCESS OF 2.5 APPEARED TO HAVE NO EFFECT ON STRENGTH OR DUCTILITY.</p> |                                            |                                                                                                                 |                             |          |
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The Effects of Thermomechanical Processing Parameters on  
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Metal Matrix Composite

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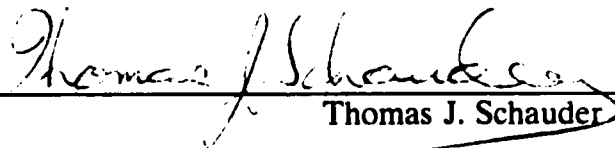
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## ABSTRACT

The effects of thermomechanical processing parameters on the elevated temperature behavior of a 6061 Al-Al<sub>2</sub>O<sub>3</sub> metal-matrix composite (MMC) have been studied. The same processing parameters were employed with unreinforced 6061 Al to provide a comparison. These materials were both thermomechanically processed at either 350°C or 500°C using two rolling schedules. Both schedules involved a constant strain per pass. Subsequent mechanical tests were conducted at temperatures 200 to 500°C and strain rates ranging from 6.7E-3 s<sup>-1</sup> to 1.31E-1 s<sup>-1</sup>. The materials processed at 500°C exhibited higher strength when compared to those processed at 350°C for deformation temperatures below 350°C. Materials stabilized by annealing after completion of rolling displayed higher ductilities when compared to the as-processed materials, especially at lower testing temperatures. The peak ductilities of the MMC's occurred at testing temperatures near the prior rolling temperatures. Solution treatment prior to rolling and additional strains during rolling in excess of 2.5 appeared to have no effect on strength or ductility.



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## I. INTRODUCTION

### A. HISTORY

Metals have useful combinations of engineering properties such as strength, ductility, and toughness. The strength of pure metals can be enhanced in many different ways, often by alloying methods and deformation processing techniques. The strength of metals can be further improved by introducing a dispersion of particles which are mechanically and thermally different from the metal itself [Ref. 1]. The size, distribution, and shape of the particles are important to the strength and performance which can be obtained.

The properties of ceramics are useful, especially at high temperatures, but they tend to suffer from very low toughness which makes them unreliable in service at ordinary temperatures. Thus, dispersing a ceramic phase in a metal matrix to form a composite can result in a combination of properties such as strength, hardness, toughness, and temperature resistance not attainable in either constituent material alone. This concept leads to metal-matrix composites (MMCs) consisting of ceramic particles or fibers in a metallic matrix [Ref. 2]. Figure 1 shows the performance regimes, temperatures and strength of several material groups. The fracture toughness of such MMCs is an especially important property for fail-safe structural use and is an area of current concern with these materials.

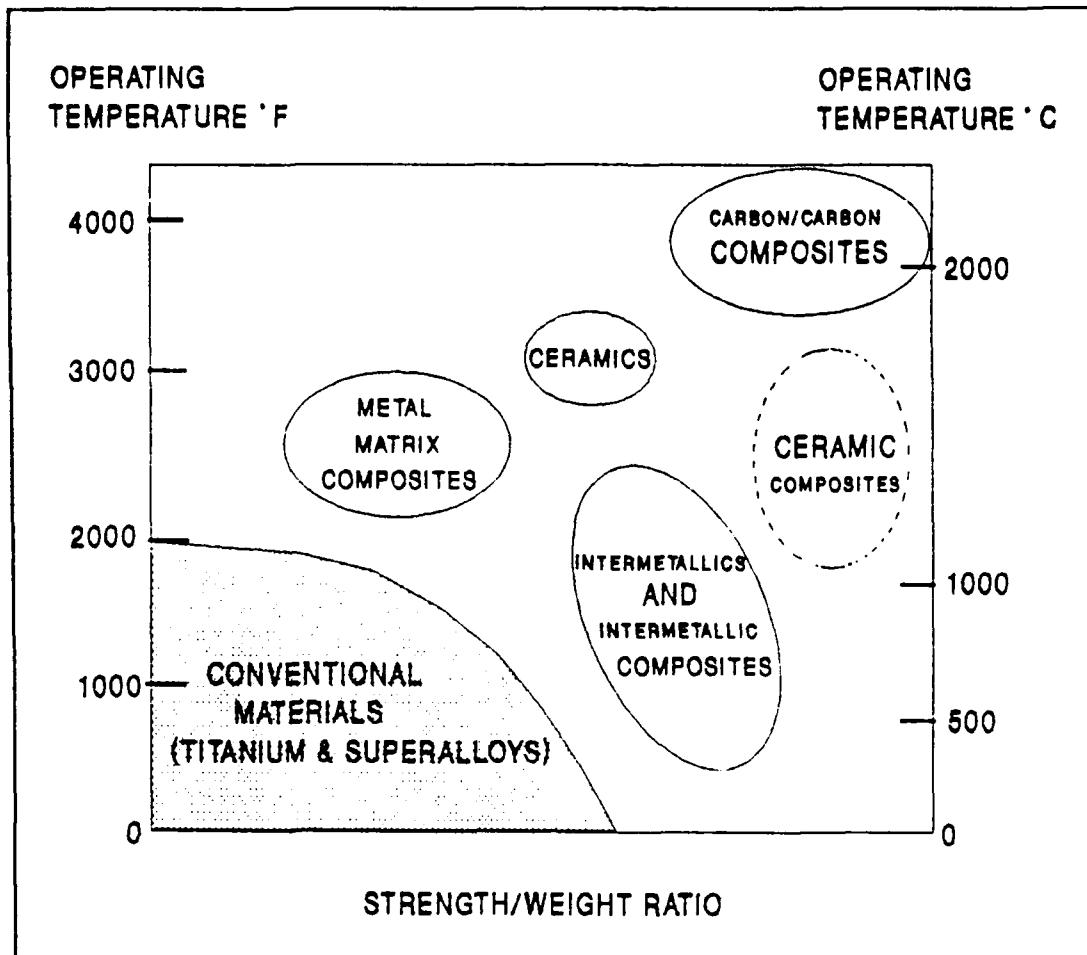


Figure 1. Strength-Temperature Map for Classes of Materials. [Ref. 2]

## B. DISCONTINUOUS METAL MATRIX COMPOSITES

A discontinuous MMC consists of hard ceramic reinforcement particles dispersed in a softer metal matrix. Other forms of reinforcement include discontinuous whiskers and continuous fibers. While such forms may result in very high strength and stiffness, properties are also highly anisotropic. Particulate additions result in potentially isotropic materials and this may offer advantages in many

applications. Advantageous properties of discontinuous MMC materials allowing significant improvements in performance of engineering components include:

- high specific modulus;
- high specific strength;
- improved fatigue and wear resistance;
- controlled thermal expansion characteristics;
- damping properties; and
- corrosion resistance.

For military systems discontinuous MMCs offer weight savings, increased flexibility in design, improved structural integrity, enhanced performance and reduced lifecycle costs. Major programs to produce lightweight, stiff, and strong metallic materials with the aid of discontinuous reinforcements have been funded. Reinforcements such as silicon carbide (SiC) and alumina ( $\text{Al}_2\text{O}_3$ ) are the ceramic materials of interest and these have melting temperatures up to  $2000^\circ\text{C}$ , considerably higher than the matrix alloy. These very hard particles are imbedded in the matrix and are generally insoluble in the matrix. For example, this results in advantageous abrasion resistance which is useful in applications such as pistons [Ref. 3]. Other applications include aircraft components, wear-resistant tooling and armor [Ref. 4]. These examples and others not specifically mentioned have sparked interest in the development of particle-reinforced, aluminum-based MMCs [Ref. 5].

## II. BACKGROUND

In aluminum-based alloys it has been demonstrated that the following variables influence the microstructure and mechanical properties of the MMC:

- the type of matrix alloy;
- the size and shape of the reinforcement;
- the volume fraction of the reinforcement;
- the relative coefficient of expansion of reinforcement and matrix; and
- the processing route for the composite.

These factors may also influence the extent of recrystallization in the matrix which also has a significant effect on MMC strength and ductility.

### A. THE MATRIX ALLOY

Aluminum and its alloys are versatile metallic materials for engineering applications. The alloy designated as 6061 constitutes the matrix of the composite studied here. It is heat treatable, of low density and good specific strength [Ref. 3]. The matrix generally is the component which limits the service temperature of the composite. The composition limits for 6061 are listed in Table I below [Ref. 6].

TABLE I. COMPOSITION LIMITS FOR 6061 AL (IN WEIGHT PERCENT)

| Si  | Fe  | Cu  | Mn   | Mg   | Cr   | Zn   | Ti   | Misc |
|-----|-----|-----|------|------|------|------|------|------|
| 0.8 | 0.7 | 0.4 | 0.15 | 0.12 | 0.35 | 0.25 | 0.15 | 0.05 |

The matrix material should effectively transmit the load to the reinforcement and should resist or stop crack propagation [Ref. 3]. Major contributing factors to the matrix alloy strength here are:

- the Peierls stress;
- the dislocation density;
- the solute content;
- precipitate size, distribution and strength; and
- grain size.

The matrix shear yield ( $\tau_y$ ) strength may be described by the equation:

$$\tau_y = \tau_p + \tau_d + \tau_s + \tau_{ppt} + \tau_{gs}$$

The Peierls stress ( $\tau_p$ ) is related to the resistance that the crystalline lattice offers to the movement of a dislocation. This value is low for an FCC metal such as aluminum. However, the added ceramic phase in a composite usually has a large value for Peierls stress and this ultimately will contribute to composite strength [Ref. 7]. The strength due to dislocations ( $\tau_d$ ) will increase as the dislocation density increases due to work hardening or other factors such as dislocation generation due to thermal expansion coefficient differences between particles and the matrix. Solute ( $\tau_s$ ) and precipitate ( $\tau_{ppt}$ ) content will also have an effect on strength. The last term is the grain size ( $\tau_{gs}$ ) contribution to strength.

In MMCs the addition of particles to form a composite may affect several of these strength terms. As the volume fraction of particles increases, dislocation density increases due to the thermal expansion differences or other factors [Ref. 8]. Such

dislocation structures may provide sites for nucleation of precipitation during heat treatment. Thus, the volume fraction and size of reinforcement particles may influence the amount of precipitation and hence the matrix strength. Therefore, such factors as alloy content, extent of precipitation, and matrix grain size must be considered in addition to the dislocation structure.

## **B. RECRYSTALLIZATION**

Recrystallization is the formation and growth of new strain free grains containing few dislocations. When a metal is heated above its recrystallization temperature, approximately  $0.4 \tau_m$ , where  $\tau_m$  is the absolute melting temperature of the metal, rapid recovery eliminates residual stresses and produces a polygonized dislocation structure. New grains then nucleate at the cell boundaries of the polygonized structure, eliminating most of the remaining dislocations. Because the number of dislocations is greatly reduced, the recrystallized metal has a low strength but a high ductility [Ref. 9].

Matrix grain size in composites can be refined due to particle stimulated nucleation (PSN) of recrystallization. The incompatibility between a deforming matrix and a non-deformable particle causes dislocations to be concentrated at the particles thereby forming a deformation zone. Large reinforcement particles stimulate nucleation owing to the formation of these deformation zones. Increased stored energy due to cold work/work hardening, higher volume percentages of the particles



and smaller interparticle spacings all contribute to a finer grain size. However, very fine particles (less than  $1\mu\text{m}$ ) and high volume fractions of these reinforcement particles can impede boundary migration therefore inhibiting recrystallization. Previous studies [Ref. 10] have shown that the as-extruded material of interest here, can achieve PSN when subjected to additional strain by thermomechanical processing using successive cycles of rolling and annealing at varying temperatures with large accumulated strains. Enhanced PSN is also attributed to larger particle size with greater spacing where nucleation sites are increased.

### **C. OTHER FACTORS AFFECTING THE MECHANICAL PROPERTIES OF DISCONTINUOUS MMCs**

The thermal expansion coefficient differences between reinforcement and the aluminum matrix alloy are significant. The coefficient of thermal expansion for aluminum is approximately four times greater than that of alumina. The thermal conductivity is reduced with the addition of ceramic particles. Properties of the resulting composite will also depend on the production method. Thermomechanical processing, such as extrusion, rolling and forging of MMCs can result in considerable strength and ductility improvements. Research on TMP techniques to improve strength and ductility have been ongoing at the Naval Postgraduate School. Previous research work on Al-Mg alloys have obtained superplastic elongations in excess of 1000% at  $300^{\circ}\text{C}$  [Ref. 11]. These rolling schedules were modified and employed to process and study ambient temperature mechanical properties of the 6061 Al- $\text{Al}_2\text{O}_3$

MMC [Ref. 12]. This research paper has been an extension and studies the effects of solution treatment, strain and TMP parameters on the elevated temperature behavior of 6061 Al and 6061 Al-Al<sub>2</sub>O<sub>3</sub> MMC.

Previous studies conducted at the Naval Postgraduate School have also cited homogeneity of particle distribution as a significant factor in attaining favorable mechanical properties in metal matrix composites. Homogenization of distribution is difficult to define, but thermomechanical processing has been performed on MMCs resulting in enhanced ductility and strength due primarily to homogeneous particle distribution. The two major factors contributing to a fine uniform microstructure are directly related to homogeneous particle distribution and uniform grain size.

The purpose of this thesis is to investigate the effect of thermomechanical processing parameters on the elevated temperature behavior of a 6061 Al-Alumina composite. The dependence of mechanical properties such as strength and ductility at elevated temperatures is also studied.

### III. EXPERIMENTAL PROCEDURE

#### A. MATERIALS AND SECTIONING

Dural Aluminum Composites Corporation provided an as-extruded 6061 Al-10 vol. pct.  $\text{Al}_2\text{O}_3$  metal-matrix composite. This material was in the form of a rectangular bar having rounded edges, with the dimensions of length 355.6mm by width 76.6mm by 19.1mm thickness (14in x 3.0in x 0.75in). Unreinforced 6061 Al was also acquired in plate form with dimensions of 381mm x 72.3mm x 25.7mm (15in x 2.9in x 1.0in).

Sectioning into billets for rolling was done with a Racine Power Hacksaw. The billets were 72.4mm x 23.9mm x 18.9mm (2.9in x 0.9in x 0.75in) and all sides were rounded to prevent cracking during subsequent rolling.

The extruded 6061 Al- $\text{Al}_2\text{O}_3$  MMC was obtained from an original cast material which has been subject to extrusion with a 17:1 area reduction [Ref. 13], corresponding to an as-extruded true strain of  $\epsilon_{ex} = 2.83$ . The nominal particle ( $\text{Al}_2\text{O}_3$ ) size was approximately  $12\mu\text{m}$ . After billets were rolled subsequent machining and tensile specimens were manufactured from the rolled strip.

#### B. THERMOMECHANICAL PROCESSING

Details of the Duralcan aluminum based discontinuous metal matrix composite processing were proprietary and not disclosed. Therefore, the first step carried out was to determine whether solution treatment at  $560^\circ\text{C}$  for 90 minutes prior to TMP

would effect the materials mechanical properties. Non-solution treated and solution treated samples were processed utilizing the same rolling schedule. Both the unreinforced 6061 Al and the 6061 Al-Al<sub>2</sub>O<sub>3</sub> MMC billets were placed in a Blue M furnace, model 8655F-3, heating the specimens to their rolling temperatures, either 350°C or 500°C, for 30 minutes prior to the first and each subsequent rolling pass. The 30 minute stabilization process allowed each billet to equilibrate at the desired rolling temperature.

The unreinforced 6061 Aluminum and the 6061 Al-Al<sub>2</sub>O<sub>3</sub> billets were rolled utilizing a Fenn Laboratory Rolling Mill and following the rolling schedules summarized in Tables II-V.

Initially, practice runs were performed to determine mill deflections. Once mill deflection was determined for the unreinforced 6061 Aluminum and the 6061 Al-Al<sub>2</sub>O<sub>3</sub> MMC, a final rolling schedule was developed. The schedule is similar to that developed here at NPS for processing of superplastic Al-Mg [Ref. 11] alloys and similar to that used for previous work on this composite [Ref. 12].

The schedule has a 10-15% reduction during the first three passes and the goal was to maintain a strain equal to 35%  $\pm$  5% for the remaining passes of the schedule. Previous studies at NPS had involved increasing strain per pass where this rolling schedule was modified to maintain a constant strain per pass in the latter passes.

The rolling strain ( $\epsilon_R$ ) shown in the tables, represents only the additional strain due to the rolling passes. This strain value ( $\epsilon_R$ ) does not include the original processing strain. For example, Table II shows the rolling strain ( $\epsilon_R$ ) equal to 2.55 after eight rolling passes. The total strain ( $\epsilon_T$ ) equals 5.38 which accounts for the strains accumulated from both of the extrusion and rolling processes.

During the rolling process, a silicone spray lubricant was used with more frequent application as the process progressed. This lubricant eliminated sticking of both the unreinforced 6061 Aluminum and the 6061 Al-Al<sub>2</sub>O<sub>3</sub> MMC during rolling. After each rolling pass the billets were placed in the furnace at their respective 350°C/500°C temperatures for 30 minutes of reheating and annealing. Small sections of the billets were cut at designated rolling passes to be polished and analyzed using optical microscopy. At the completion of the final rolling pass the rolled material was quenched in water to ambient temperature. Approximately half the material obtained was left in the as-processed condition. The remaining half of the rolled strip was placed in the furnace to anneal 30 minutes for stabilization. Tensile specimens were then prepared for further study.

**TABLE II. ROLLING SCHEDULE FOR 6061 Al PROCESSED AT 350°C**

| ROLL #              | $T_o$  | $T_i$  | MILL GAP (IN) | DEFLECTION | STRAIN |
|---------------------|--------|--------|---------------|------------|--------|
| 1                   | 0.7465 | 0.6585 | 0.64          | 0.0185     | 0.118  |
| 2                   | 0.6585 | 0.588  | 0.57          | 0.018      | 0.107  |
| 3                   | 0.588  | 0.5215 | 0.5           | 0.0215     | 0.113  |
| 4                   | 0.5215 | 0.3565 | 0.335         | 0.0215     | 0.3163 |
| 5                   | 0.3565 | 0.217  | 0.195         | 0.022      | 0.3913 |
| 6                   | 0.217  | 0.1405 | 0.117         | 0.0235     | 0.3525 |
| 7                   | 0.1405 | 0.0915 | 0.068         | 0.0235     | 0.3487 |
| 8                   | 0.0915 | 0.058  | 0.034         | 0.024      | 0.366  |
| $\epsilon_R = 2.55$ |        |        |               |            |        |
| 9                   | 0.058  | 0.0355 | 0.013         | 0.023      | 0.3879 |
| $\epsilon_R = 3.05$ |        |        |               |            |        |

TABLE III. ROLLING SCHEDULE FOR 6061 Al PROCESSED AT 500°C

| ROLL # | T <sub>0</sub> | T <sub>1</sub> | MILL GAP (IN) | DEFLECTION | STRAIN              |
|--------|----------------|----------------|---------------|------------|---------------------|
| 1      | 0.7465         | 0.657          | 0.64          | 0.017      | 0.119               |
| 2      | 0.657          | 0.582          | 0.57          | 0.012      | 0.114               |
| 3      | 0.582          | 0.518          | 0.5           | 0.018      | 0.109               |
| 4      | 0.518          | 0.351          | 0.335         | 0.016      | 0.322               |
| 5      | 0.351          | 0.2365         | 0.2195        | 0.017      | 0.3263              |
| 6      | 0.2365         | 0.1515         | 0.135         | 0.0165     | 0.359               |
| 7      | 0.1515         | 0.099          | 0.08          | 0.019      | 0.347               |
| 8      | 0.099          | 0.065          | 0.043         | 0.022      | 0.343               |
|        |                |                |               |            | $\epsilon_R = 2.44$ |
| 9      | 0.065          | 0.04           | 0.018         | 0.022      | 0.385               |
|        |                |                |               |            | $\epsilon_R = 2.93$ |

TABLE IV. ROLLING SCHEDULE FOR 6061 Al-10  
VOL. PCT.  $\text{Al}_2\text{O}_3$  PROCESSED AT 350°C

| ROLL # | $T_o$  | $T_i$  | MILL GAP (IN) | DEFLECTION | STRAIN              |
|--------|--------|--------|---------------|------------|---------------------|
| 1      | 0.7545 | 0.688  | 0.67          | 0.018      | 0.088               |
| 2      | 0.69   | 0.6165 | 0.6           | 0.017      | 0.107               |
| 3      | 0.6165 | 0.5435 | 0.53          | 0.014      | 0.118               |
| 4      | 0.5435 | 0.378  | 0.356         | 0.022      | 0.304               |
| 5      | 0.378  | 0.2575 | 0.238         | 0.0195     | 0.319               |
| 6      | 0.2575 | 0.169  | 0.151         | 0.018      | 0.343               |
| 7      | 0.169  | 0.1055 | 0.085         | 0.0205     | 0.376               |
| 8      | 0.1055 | 0.0655 | 0.045         | 0.0205     | 0.379               |
|        |        |        |               |            | $\epsilon_R = 2.44$ |
| 9      | 0.0655 | 0.0395 | 0.017         | 0.0225     | 0.397               |
|        |        |        |               |            | $\epsilon_R = 2.95$ |



TABLE V. ROLLING SCHEDULE FOR 6061 Al-10  
VOL. PCT.  $\text{Al}_2\text{O}_3$  PROCESSED AT 350°C

| ROLL # | $T_o$  | $T_f$  | MILL GAP (IN) | DEFLECTION | STRAIN              |
|--------|--------|--------|---------------|------------|---------------------|
| 1      | 0.7545 | 0.69   | 0.67          | 0.02       | 0.085               |
| 2      | 0.69   | 0.618  | 0.6           | 0.018      | 0.104               |
| 3      | 0.618  | 0.55   | 0.53          | 0.02       | 0.11                |
| 4      | 0.55   | 0.3785 | 0.36          | 0.0185     | 0.311               |
| 5      | 0.3785 | 0.257  | 0.242         | 0.015      | 0.321               |
| 6      | 0.257  | 0.176  | 0.154         | 0.022      | 0.315               |
| 7      | 0.176  | 0.119  | 0.1           | 0.019      | 0.324               |
| 8      | 0.119  | 0.08   | 0.0595        | 0.0205     | 0.328               |
|        |        |        |               |            | $\epsilon_R = 2.24$ |
| 9      | 0.08   | 0.055  | 0.03          | 0.025      | 0.313               |
|        |        |        |               |            | $\epsilon_R = 3.22$ |

### C. MACHINING

Processed material representing all four conditions: Unreinforced 6061 Al, as-processed and stabilized; 6061 10 vol. pct.  $\text{Al}_2\text{O}_3$  MMC; as-processed and stabilized, were machined to the dimensions for tensile testing as shown in Figure 2. Tensile specimens were machined using a cobalt cutting tool with 5/8" four-lip end mills, 1/16" radius.

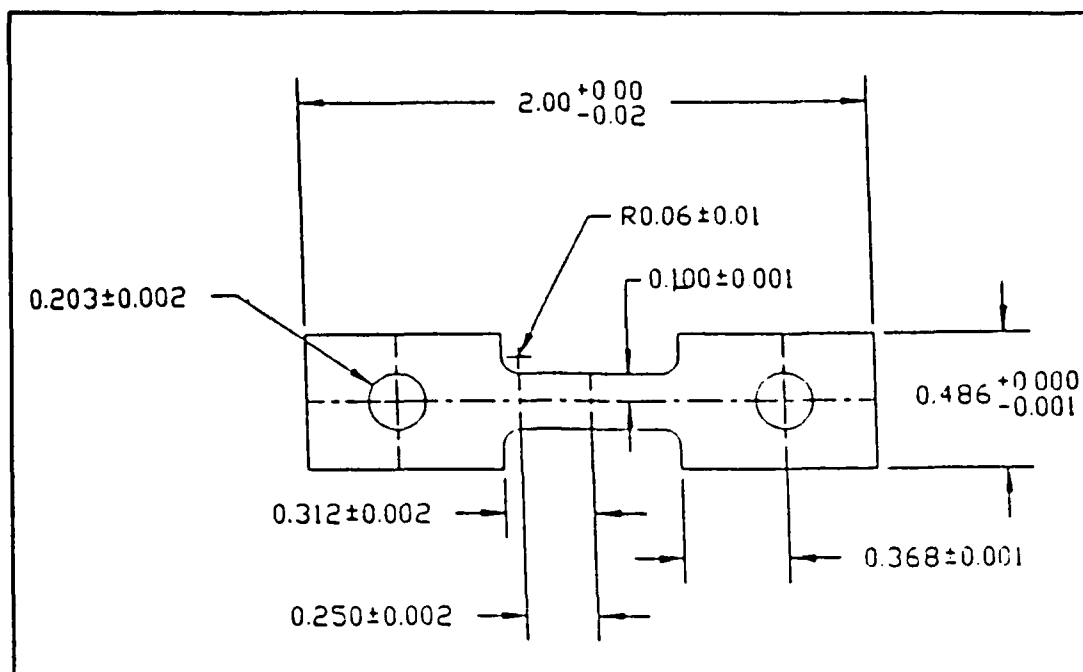


Figure 2. Tensile Test Specimen Drawing

#### D. TENSILE TESTING

Tensile testing was performed on an Instron Model 6027 testing machine. A Marshall tubular furnace (1200°C, 60 Hz, 110V) was mounted on the Instron and provided temperature control with an accuracy of  $\pm 4^\circ\text{C}$ . Computer interface with an Instron 6000 series control console and plotter provided stress vs. strain, load vs. displacement as well as peak strength information. Six thermocouples were initially mounted inside the furnace to monitor the temperature gradient and to establish the actual time for the tensile specimen to reach testing temperature.

The temperature gradient was adjusted utilizing 3 and 4 ohm shunts, but the no-shunt condition proved to be adequate as seen in Table VI. Several test runs were

conducted to establish the time for a tensile specimen to reach testing temperature and the plot is shown in Figure 3. Samples were given 45 minutes to equilibrate and then held at temperature for 5 minutes prior to each test. The temperature of the sample was within 3-5 degrees of desired test temperature.

Tensile tests were conducted utilizing crosshead speeds varying from 5.1 mm/min to 100.0 mm/min, providing strain rates of  $6.7\text{E-}3\text{s-}1$ ,  $6.7\text{E-}2\text{s-}1$ , and  $1.3\text{E-}1\text{s-}1$ . The test temperatures ranged from  $200^{\circ}\text{C}$  to  $550^{\circ}\text{C}$ .

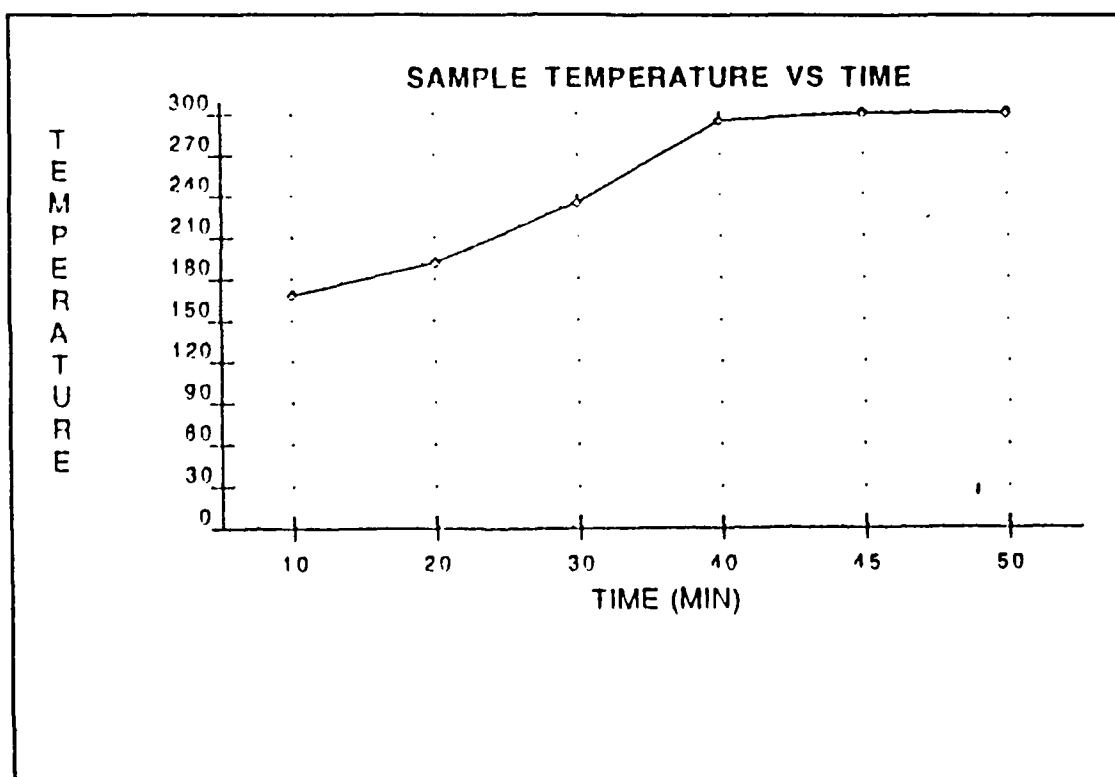


Figure 3. Time for Tensile Sample to Reach Testing Temperature

**TABLE VI. FURNACE SHUNT TABLE**

|    | A            | B           | C           | D                 | E        |
|----|--------------|-------------|-------------|-------------------|----------|
| 1  | THERMOCOUPLE | 3 OHM SHUNT | 4 OHM SHUNT | 3 AND 4 OHM SHUNT | NO SHUNT |
| 2  | (TEMP C)     | (ZONE 3-4)  | (ZONE 3-4)  | (ZONE 3-4/4 OHM)  |          |
| 3  |              |             |             | (ZONE 5-6/3 OHM)  |          |
| 4  |              |             |             |                   |          |
| 5  | 1            | 301         | 305         | 300               | 309      |
| 6  | 2            | 310         | 305         | 301               | 309      |
| 7  | 3            | 315         | 316         | 314               | 314      |
| 8  | 4            | 301         | 314         | 312               | 310      |
| 9  | 5            | 300         | 311         | 310               | 309      |
| 10 | 6            | 302         | 303         | 300               | 300      |

**E. DATA REDUCTION**

Stress vs. strain data and load vs. displacement curves were obtained via computer programs using standard methods. Grip slippage was observed in many tests and was eliminated only by preloading the tensile specimen. To compensate for the grip slippage, the equation of the line was fitted to the elastic region of the load vs. displacement curve. This line was transposed parallel to the point of fracture and the distance was then measured between the two lines. Calculations of strain were then documented and the results plotted.

## F. MICROSCOPY

Optical microscopy was conducted utilizing a Zeiss ICM-405 microscope. Samples were mounted and polished using procedures similar to those of previous work [Ref. 12]. Sample polishing procedures were accomplished as outlined in Table VII. The goal was to achieve a light background matrix with the dark contrast alumina particles. Too little polishing resulted in excess scratches. Over polishing with excess diamond paste obscured the clarity under the objective and resulted in diamond paste particulates embedding themselves into the matrix. Using very little diamond paste and changing the selvyt cloth frequently was the key to obtain clean, well polished samples.

The scanning electron microscope using secondary electron imaging methods was used to study the fracture modes of the unreinforced 6061 Al and the 6061 Al-Al<sub>2</sub>O<sub>3</sub> MMC.

**TABLE VII. SAMPLE POLISHING PROCEDURES**

| <b>STEP #</b> | <b>POLISHING MEDIUM</b>         | <b>TIME (MIN)</b>    | <b>WHEEL RPM</b> | <b>COMMENTS</b>             |
|---------------|---------------------------------|----------------------|------------------|-----------------------------|
| 1             | 320 Grit                        | 2                    | 180-200          | Light pressure              |
| 2             | 400 Grit                        | 2                    | 180-200          | Light pressure              |
| 3             | 600 Grit                        | 2                    | 180-200          | Light pressure              |
| 4             | 6 Micron diamond paste (metadi) | 3                    | 180-200          | Light pressure              |
| 5             | 3 Micron diamond paste (metadi) | 2                    | 180-200          | Light pressure              |
| 6             | Colloidal Silica                | Remove all scratches | 180-200          | Light pressure, wear gloves |

## IV. RESULTS

### A. INFLUENCE OF PROCESSING ON MICROSTRUCTURE

The effect of rolling strain on microstructure was studied using optical microscopy methods to investigate the evolution of the  $\text{Al}_2\text{O}_3$  particle distribution during processing of the MMC. Figure 4 shows a typical example of this evolution. Figures 4a and 4b presents optical micrographs of materials rolled at 350°C and 500°C after three passes. These materials have experienced rolling strains around 0.32, and total accumulated strains  $\epsilon_T = 3.1$  ( $\epsilon_T = \epsilon_{ex} + \epsilon_R$ ). These figures also show varying degrees of clustering and banding. Figures 4c and 4d are micrographs representative of the MMC rolled at 350°C and 500°C through 9 passes. These materials have experienced rolling strains around 3.0, and total accumulated strains of 5.83 ( $\epsilon_T = 5.83$ ). The additional induced strain has resulted in a more homogeneous structure, although alignment of particles in the rolling direction and very fine clusters are still evident in the micrographs.

### B. SENSITIVITY TO PROCESSING VARIATIONS

#### 1. Solution Treatment vs. Non-Solution Treatment

As mentioned earlier, details of the Duralcan aluminum based discontinuous metal matrix composite processing were proprietary and not disclosed. Therefore, the first step carried out was to determine whether solution treatment at 560°C for 90 minutes prior to TMP would effect the materials mechanical properties. Figure 5 is a plot of ductility vs. test temperature for MMC material rolled at 500°C. These data are nearly identical and

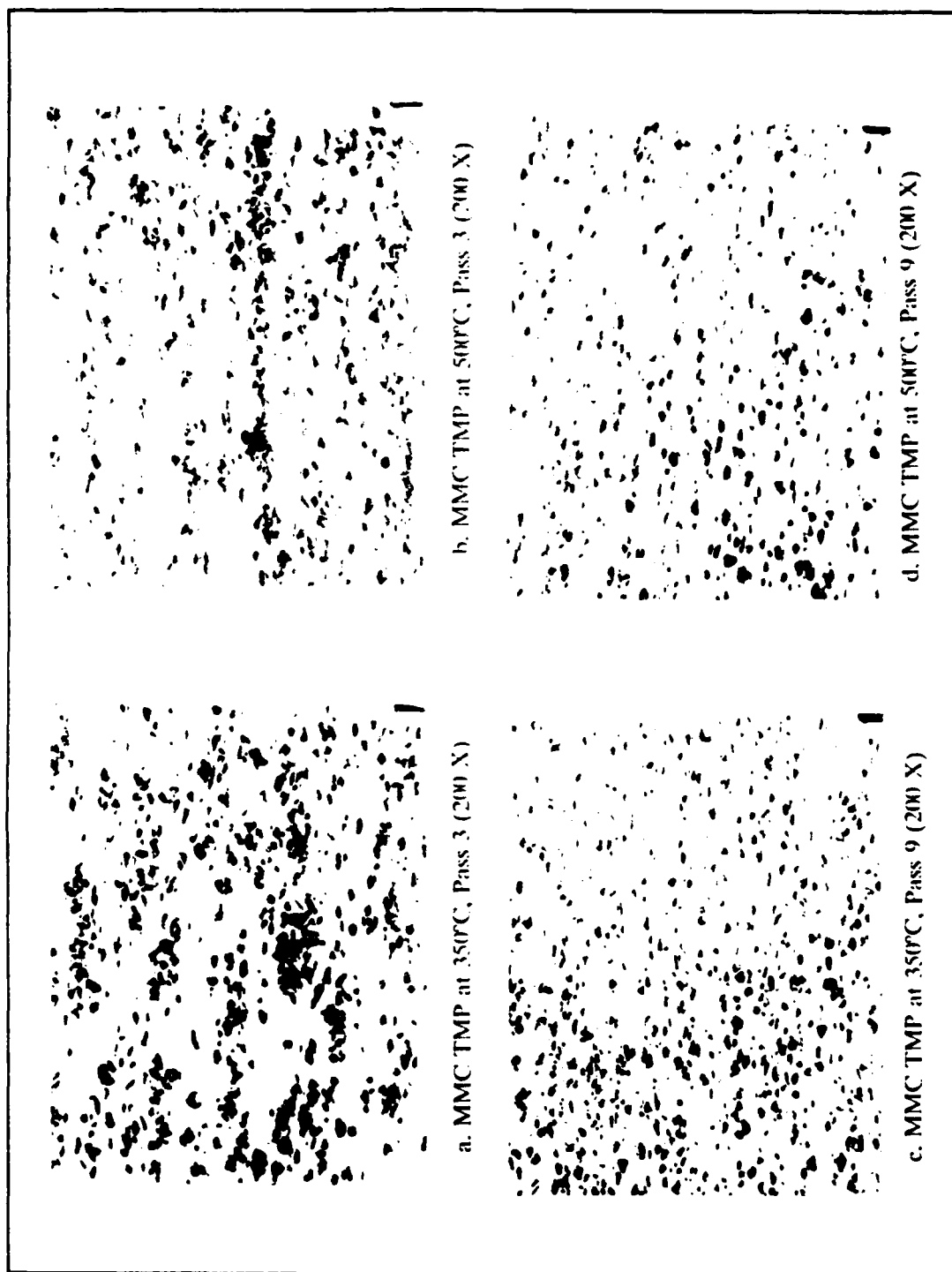


Figure 4. Micrographs for MMCs Thermomechanically Processed at 350°C and 500°C Showing Passes 3 and 9



that solution treatment has no effect on ductility and strength for this material. These data also show increasing ductility with increasing test temperatures, with ductility attaining approximately 50% elongation to failure at the end of tests. Figure 6 is a plot of ultimate tensile strength (UTS) data vs. test temperature corresponding to ductility data in Figure 5. Again, the solution treatment process has no significant effect on strength or ductility. This composite material exhibits a rapid decrease in strength with temperatures up to 400°C and a slower decrease at the higher test temperatures. Similar results were obtained for ductility and strength responses with the other processed materials.

## 2. Total Processing Strain ( $\epsilon_R = 2.2$ vs. $\epsilon_R = 3.2$ )

The effect of total strain was also evaluated by mechanical testing. Figure 7 is a plot of ductility vs. test temperature, and Figure 8 is a plot of UTS vs. temperature for materials processed identically except one case  $\epsilon_R = 2.2$  and the other case  $\epsilon_R = 3.2$ . Again, comparison of these figures shows no discernable difference in ductility vs. temperature behavior or UTS vs. temperature is attributable to the difference in processing strain. Note that Figures 7 and 8 use the same data for the corresponding solution treated plot and the plot for rolling strain = 3.2 in sections 1 and 2.

### 3. Effect of Stabilization on Mechanical Response

#### a. *MMC Materials Processed at 350°C*

As mentioned earlier, previous studies conducted at the Naval Postgraduate School have shown homogeneity of particle distribution to be a significant factor in the mechanical properties of MMCs. PSN of recrystallization occurs during the rolling process and 30 minute anneals between passes. A resulting reduction in the MMC matrix grain size along with improved particle distribution corresponds to increased ductility. This work assesses the mechanical response on as-processed and stabilized MMCs.

Figure 9 is a plot of ductility vs. temperature for MMC as-processed material rolled at 350°C and tested at various strain rates. These data show identical trends indicating the ductility is relatively low up to temperatures of 200°C and then increases rapidly from 200-400°C, but then drops off significantly at still higher temperatures. Figure 10 is a plot of ductility vs. temperature for MMC material rolled at 350°C and stabilized by annealing 30 minutes at this temperature. This was conducted at two different strain rates. Comparison of Figures 9 and 10 reveals higher ductility at lower test temperatures for the stabilized material. This is expected and is directly attributable to the occurrence of recovery and possible recrystallization during the stabilization process. The dislocation density is decreased and the associated ductility is increased. Figures 11 and 12 are plots of the UTS vs. temperature for the as-processed and stabilized MMC materials tested at various

strain rates. Comparison of these plots show the as-processed MMC has higher UTS values up to test temperatures of 350°C, around the rolling temperature, after which the strengths are similar and continue to decrease with further increases in temperatures.

*b. Unreinforced 6061 Al Processed at 350°C*

A similar analysis concerned with the effect of stabilizing and anneal was done with unreinforced 6061 Al. Figure 13 is a plot of ductility vs. temperature for 6061 Al as-processed material rolled at 350°C and tested at two different strain rates. These data show similar trends indicating ductility is relatively low up to temperatures of 200°C and increases rapidly from 200-500°C. Figure 14 is a plot of ductility vs. temperature for 6061 Al stabilized material rolled at 350°C and tested at two different strain rates. This figure shows higher ductility at lower test temperatures. The comparison of these two figures shows the stabilization and anneal at the conclusion of rolling again results in substantial increases in ductility at lower temperatures. Figures 15 and 16 are plots of the UTS vs. temperature for the as-processed and the stabilized 6061 Al materials tested at two different strain rates. Comparison of these plots show the as-processed 6061 Al has higher UTS values up to test temperatures around the 350°C rolling temperature, after which the strengths are similar and continue to decrease. These data show exactly the same trend as that of the MMC material. Identical processing methods used on unreinforced 6061 Al

and MMC materials have given the same UTS results, mostly apparent at lower test temperatures.

Ductility for the unreinforced 6061 Al is always similar to or greater than that of the MMC. Elongations for the unreinforced 6061 Al reached maximums of approximately 110% compared to approximately 70% for the MMC. Therefore, with the homogenization of the particle distribution, the ductility of the MMC is not seriously degraded. The stabilized condition is more ductile for test temperatures up to 350°C. Above 350°C test temperatures the processed and stabilized materials have similar elongation characteristics.

*c. MMC Materials Processed at 500°C*

Figures 17 and 18 are plots of ductility vs. temperature for MMC as-processed and stabilized materials rolled at 500°C and tested at various strain rates. These two figures show that stabilized materials at 500°C rolling temperatures have a 20% ductility compared to 10% for the as-processed MMCs, at lower temperatures. The as-processed and stabilized materials have similar ductilities, around 50% at higher temperatures, where these materials are not as sensitive to temperature. Results also show an increase in ductility with faster strain rates (i.e.  $1.3\text{E-}1\text{ s}^{-1}$ ) and this ductility is maintained at higher temperatures.

Figures 19 and 20 are plots of the UTS vs. temperature for the as-processed and stabilized MMC materials tested at various strain rates. Comparison of these plots show the as-processed MMC has higher UTS values up to test

temperatures of 350°C, after which the strengths are similar and continue to decrease. Results also show that the faster strain rates do not have any effect on strength, especially at temperatures above 400°C.

The as-processed and stabilized MMC materials rolled at 500°C exhibit similar trends in their respective ductility data. However, when comparing these materials to those rolled at 350°C, the peak ductility is lower. For example, Figures 7 and 9 are ductility vs. temperature plots for the as-processed MMCs rolled at 350°C and 500°C, respectively, Figure 7 shows the MMC processed at 500°C reaches its peak ductility of 55% at approximately 400°C, and maintains this ductility at higher temperatures. Figure 9 shows that the MMC processed at 350°C reaches a peak ductility of 75% at 400°C, but then declines significantly upon further heating. This implies that the material processed at 500°C is less dependent on subsequent annealing due to the higher rolling temperature. The data also shows the higher strain rates resulted with increased ductility.

UTS data collected for the same materials, processed at their respective 350°C and 500°C temperatures, shows that the material processed at 500°C is consistently stronger than the 350°C processed material. These results were surprising, as higher strengths were expected of the materials processed at 350°C. Larger dislocation densities are usually generated due to lower processing temperatures which would contribute to the total strength of the matrix alloy. However, these results imply that the additional effects of strengthening due to

precipitates and solute may be significant and are not taken into account with processing temperature.

*d. Unreinforced 6061 Al Processed at 500°C*

Figures 21 and 22 are plots of ductility vs. temperature for 6061 Al as-processed and stabilized 6061 material rolled at 500°C and tested at two different strain rates. Comparison of these two figures shows that stabilized materials again exhibit higher ductility at lower temperatures. At the higher testing temperatures the stabilization effects are not as apparent due primarily to the rapid recovery within the alloy. Figures 23 and 24 are corresponding plots of the UTS vs. temperature for these materials. UTS vs. temperature data shows similar trends especially at higher test temperatures. At lower temperatures the effect of processing is more apparent, the stabilized material has a lower strength at the lower temperatures.

The data consistently reveals that the MMC and unreinforced 6061 Al materials processed at 500°C were stronger at lower temperatures when compared to the 350 C material. For all cases the as-processed materials had higher UTS strengths than the stabilized materials up to 400°C test temperatures. Above 400°C testing temperatures all materials, processed and stabilized, display similar UTS characteristics.

### C. SEM/FRACTOGRAPHY

The scanning electron microscope using secondary electron imaging methods was used to study the fracture modes of the unreinforced 6061 Al and the 6061 Al-Al<sub>2</sub>O<sub>3</sub> MMC. Figures 25a and 25b show fractographs of the MMCs thermomechanically processed at the two corresponding temperatures of 350°C and 500°C. These figures illustrate effects of processing temperature on fracture mode at a low test temperature of 200°C. It is apparent that there is no distinguishable difference between the two fractographs. Both samples show microvoid formation and coalescence.

Figures 25 and 26 show the effects of test temperature on the MMC fracture mode. Although the MMC material was generally more ductile at higher test temperatures, microvoid formation is less apparent at these higher test temperatures, dimples are shallower and the fracture appears faceted. However, grain boundary sliding may be occurring which may be a reason for this increased ductility.

Figure 27 illustrates the role of particles in void formation. An alumina particle is seen residing at the base of a microvoid. The alumina particles act as a stress concentration causing a separation which results in microvoid formations. Figure 28 presents fractographs for the unreinforced 6061 Al for two different processing conditions. The unreinforced matrix material shows void formation and coalescence is the predominant mode for fracture at both 200°C and 500°C. Comparison of these modes with earlier figures indicate similar trends in that both the MMC and

unreinforced 6061 Al material fail by void formation and coalescence at lower test temperatures.



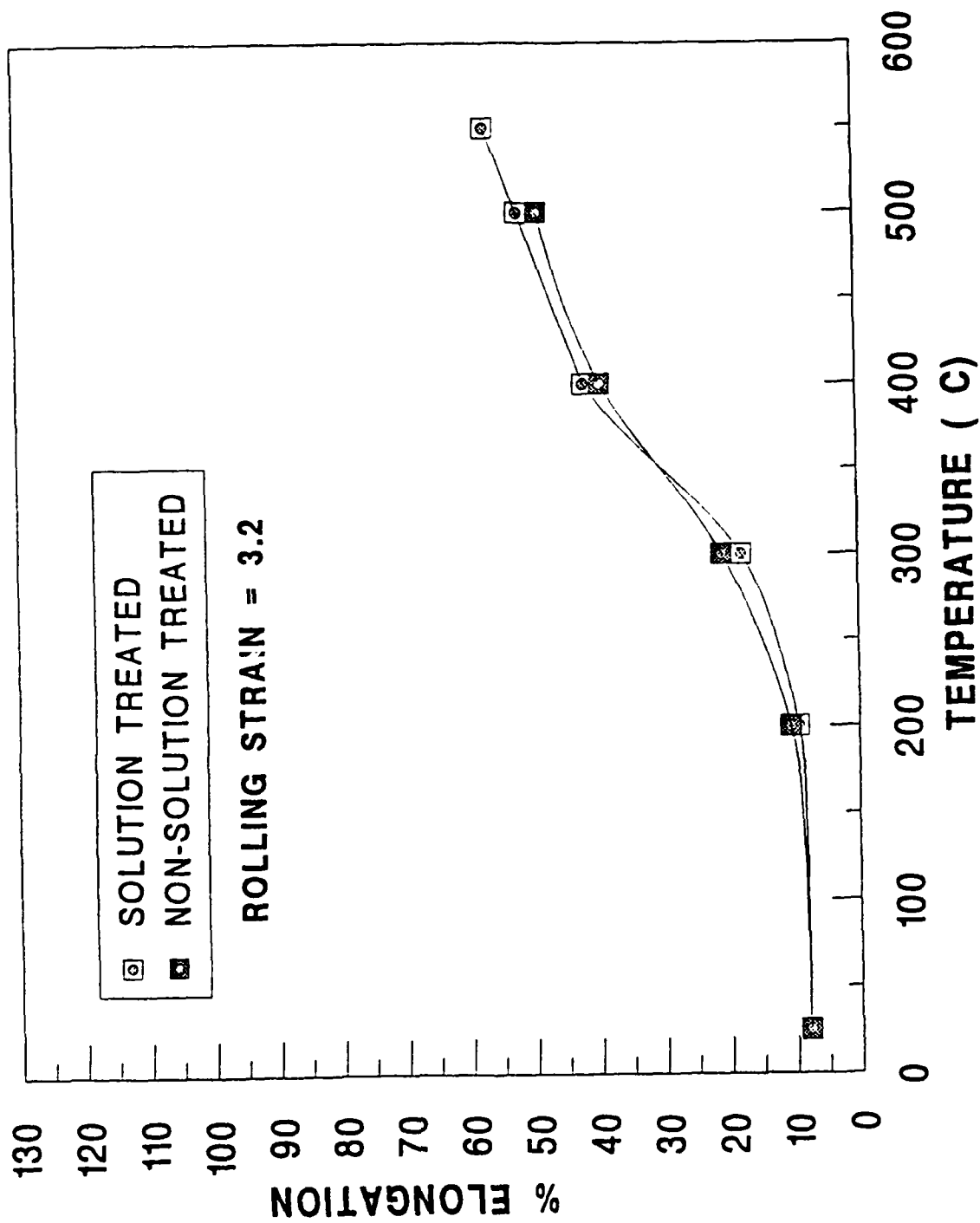


Figure 5. Effects of Solution Treatment on Ductility of an As-processed MMC Rolled at 500°C (Strain Rate =  $6.7 \text{ E-}2 \text{ s}^{-1}$ )

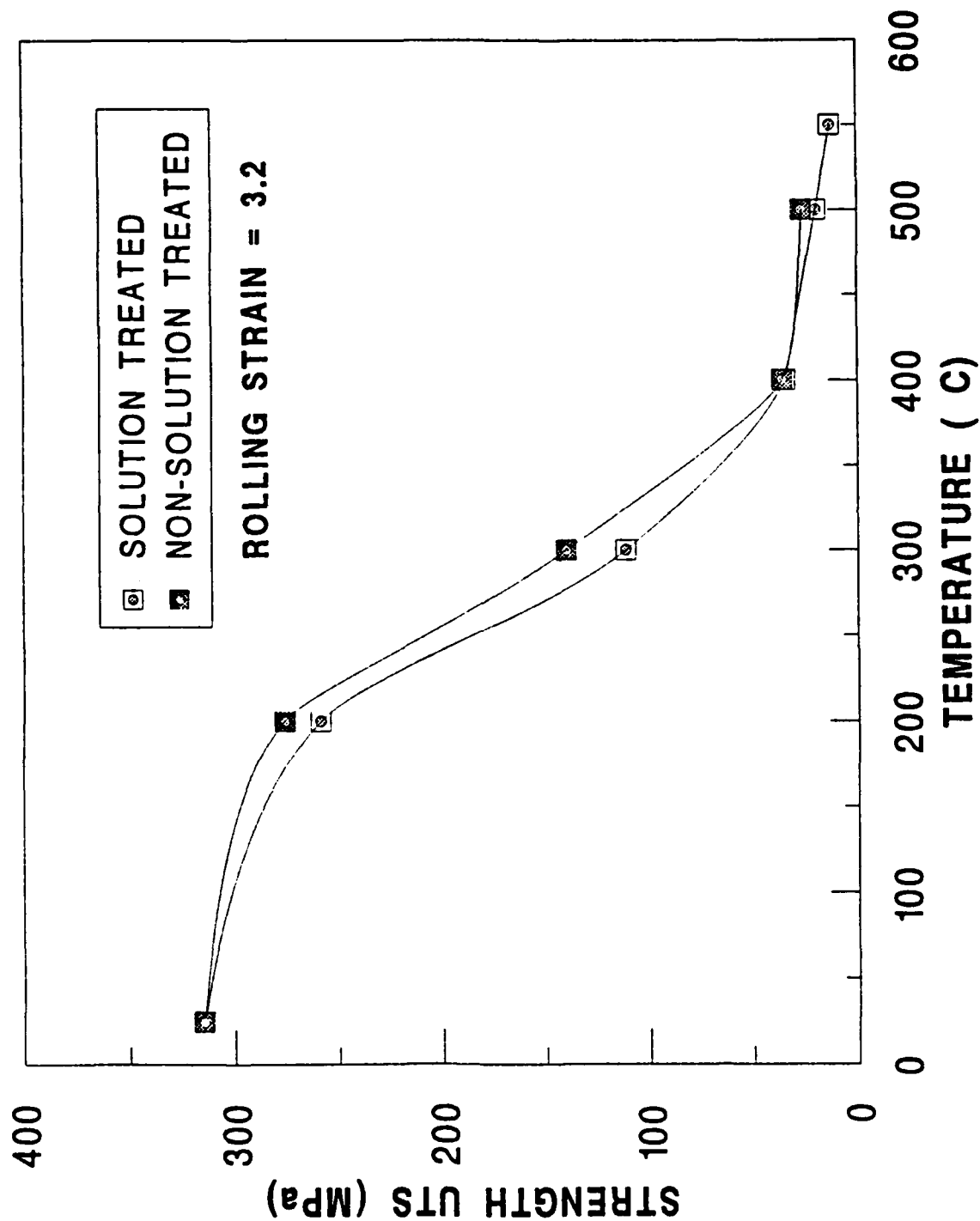


Figure 6. Effect of Solution Treatment on UTS of an As-processed MMC Rolled at 500°C (Strain Rate =  $6.7 \times 10^{-2} \text{ s}^{-1}$ )

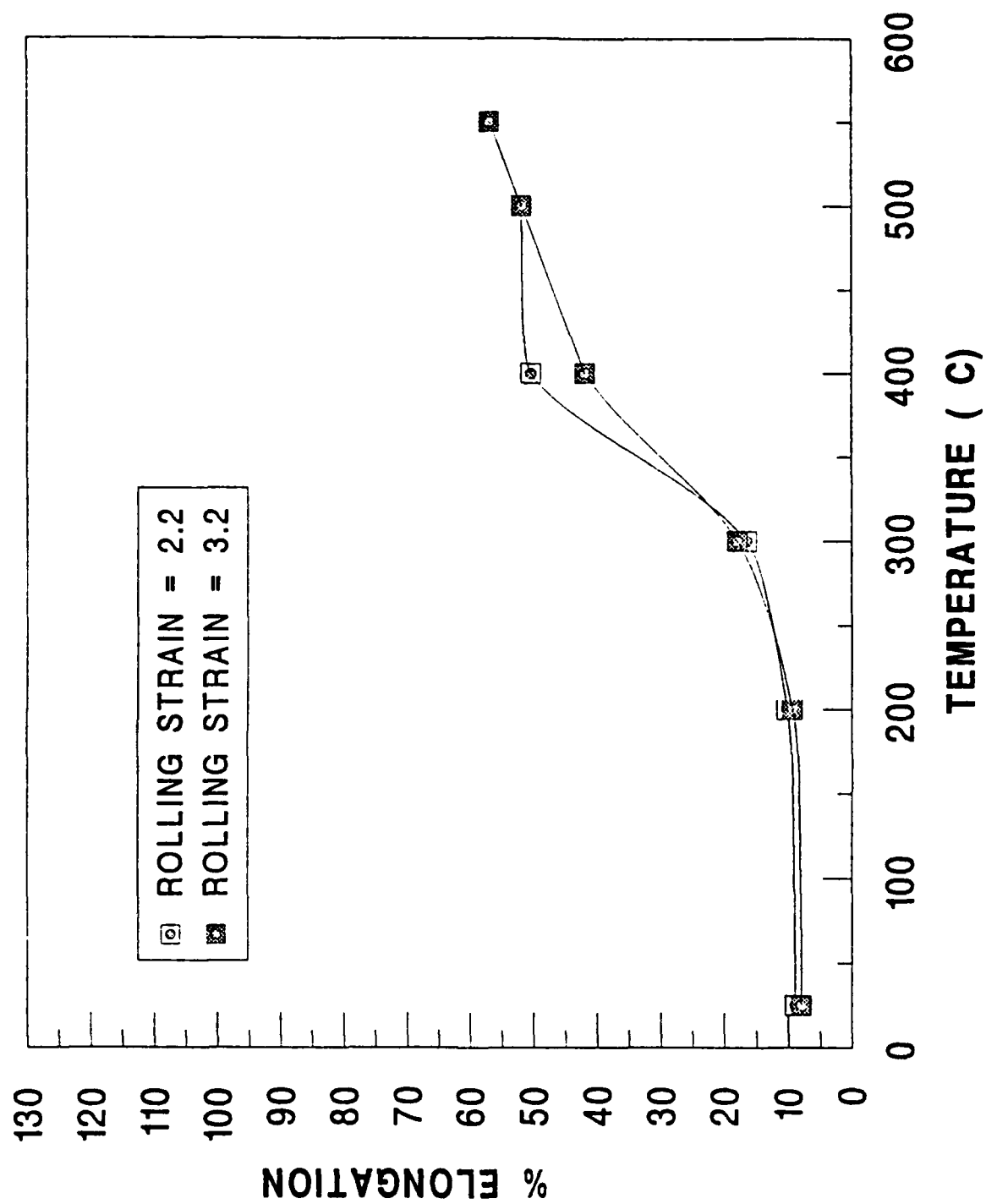


Figure 7. Effects of Total Processing Strain on Ductility for an As-processed MMC Rolled at 500°C (Roll Pass 8 vs. Roll Pass 9)

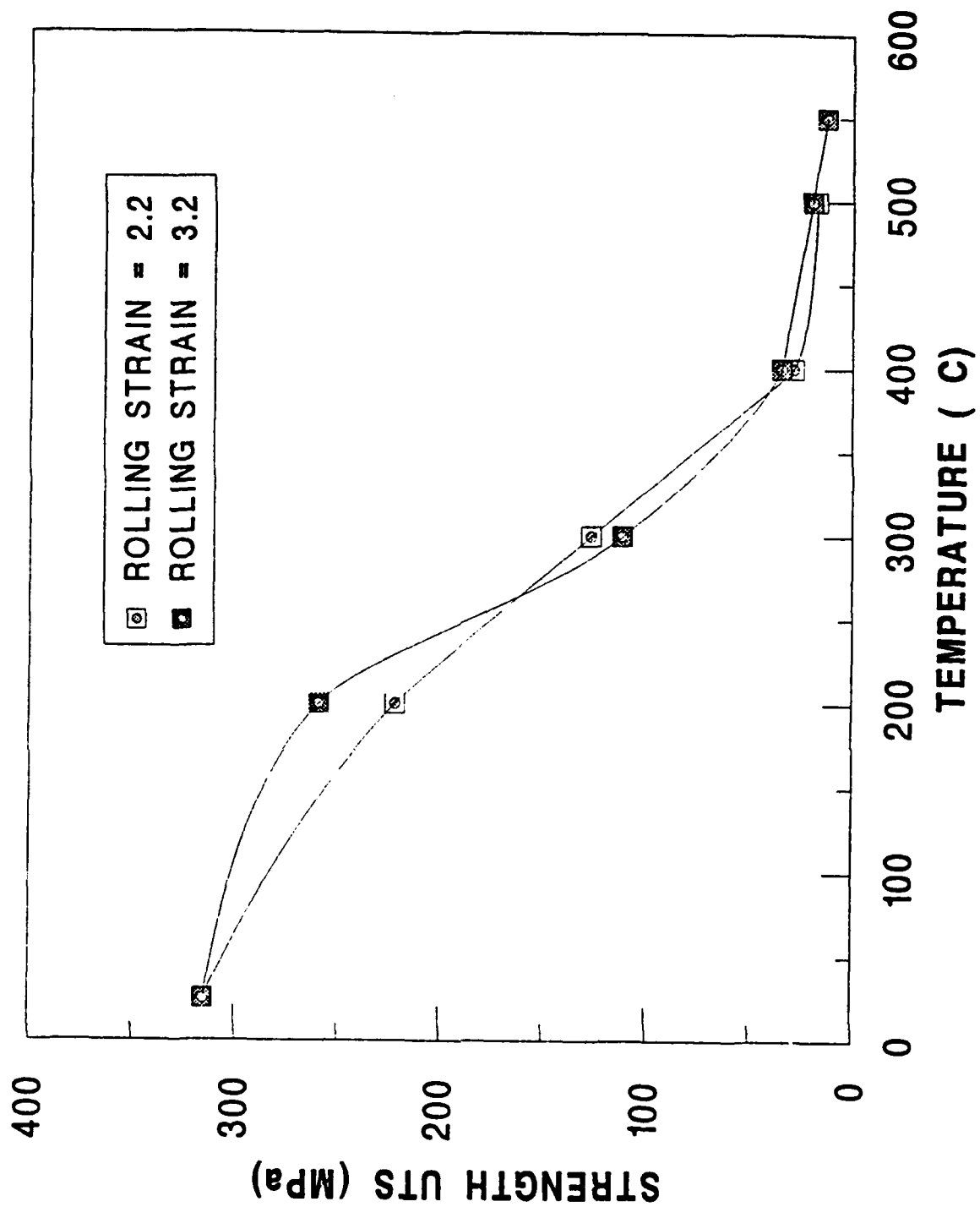


Figure 8. Effects of Total Processing Strain on UTS for an As-processed MMC Rolled at 500°C (Roll Pass 8 vs. Roll Pass 9)

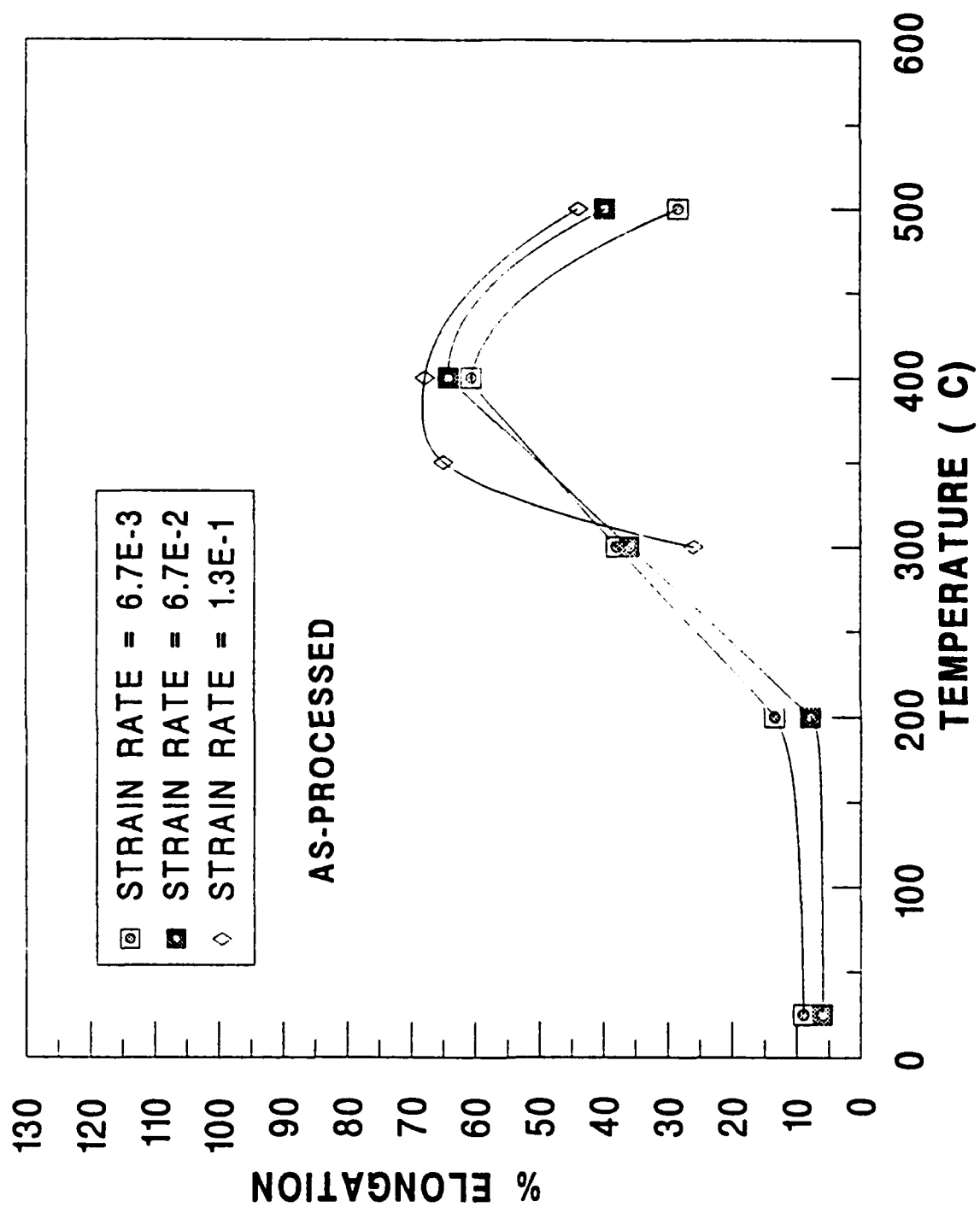


Figure 9. Ductility vs. Temperature for an As-processed MMC Rolled at 350°C

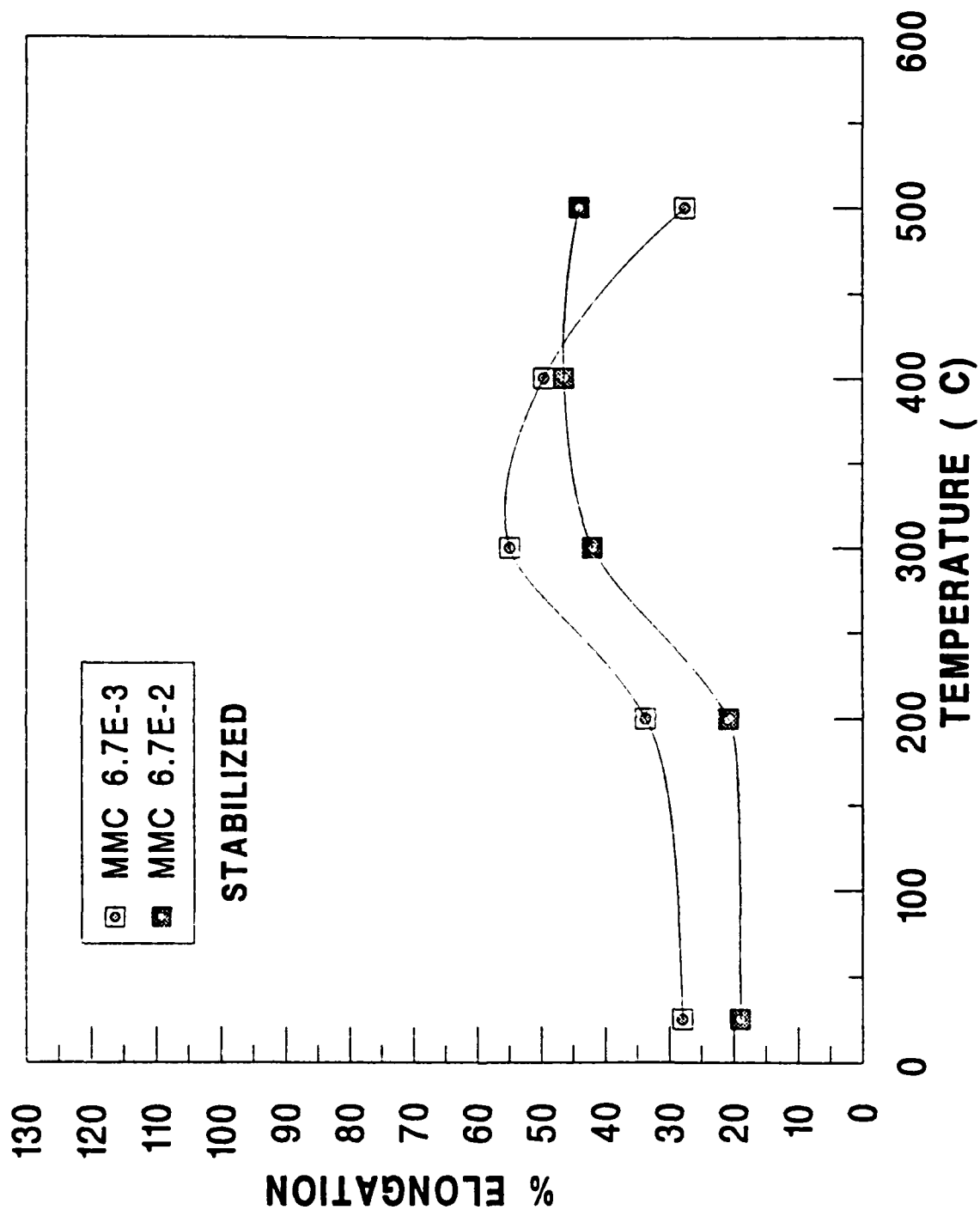


Figure 10. Ductility vs. Temperature for a Stabilized MMC Rolled at 350°C

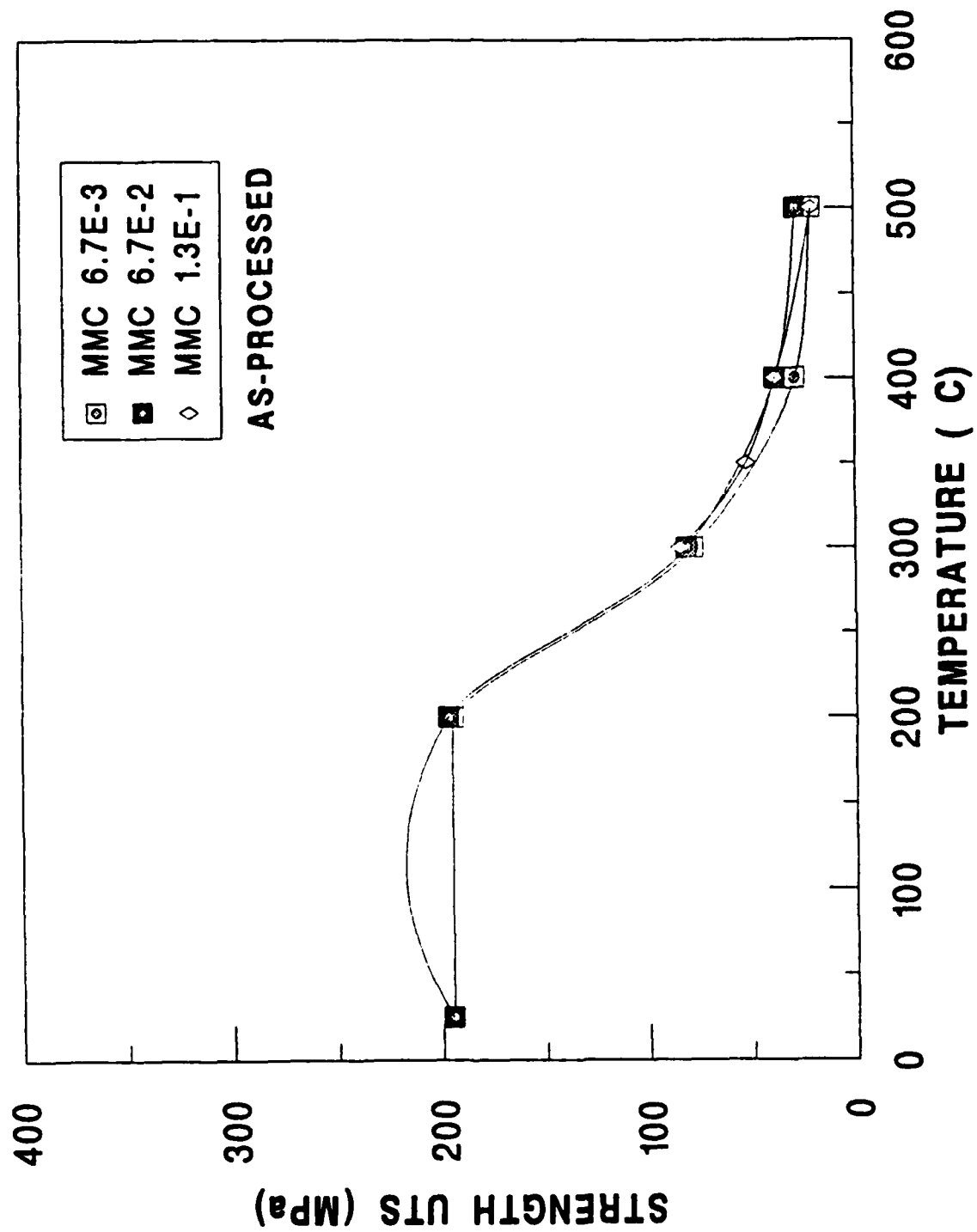


Figure 11. UTS vs. Temperature for an As-processed MMC Rolled at 350°C

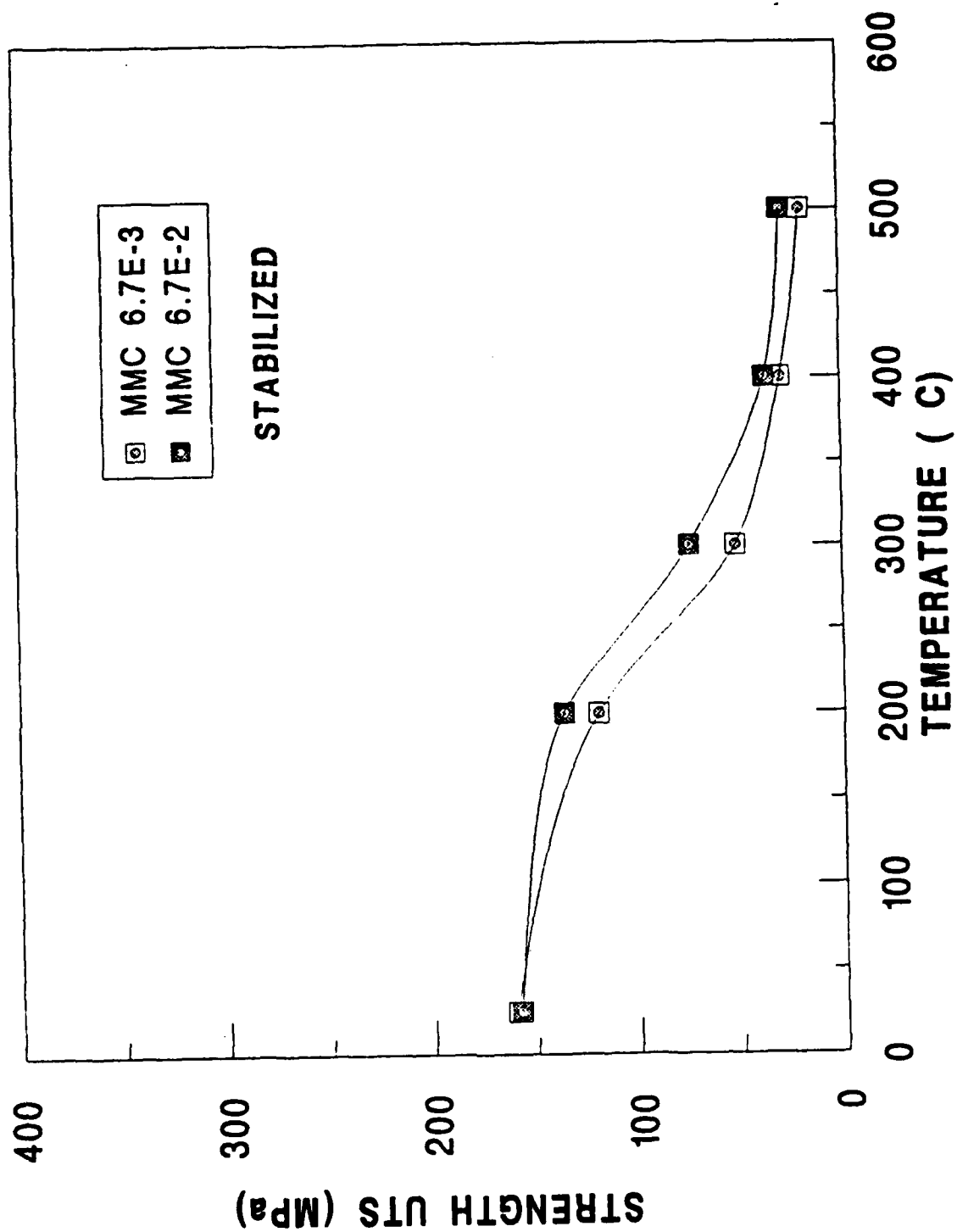


Figure 12. UTS vs. Temperature for a Stabilized MMC Rolled at 350°C



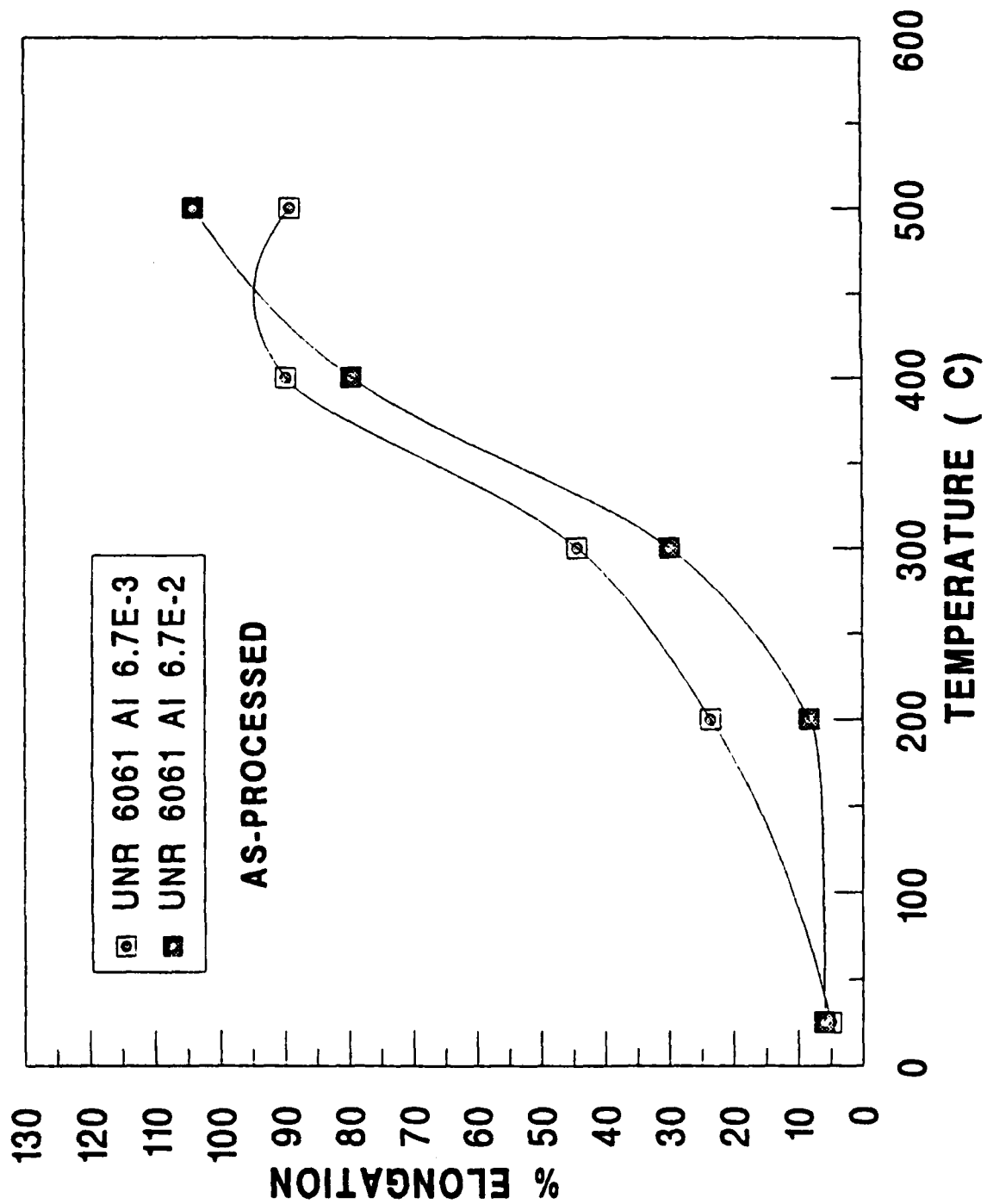


Figure 13. Ductility vs. Temperature for an As-processed Unreinforced 6061 Al Rolled at 350°C

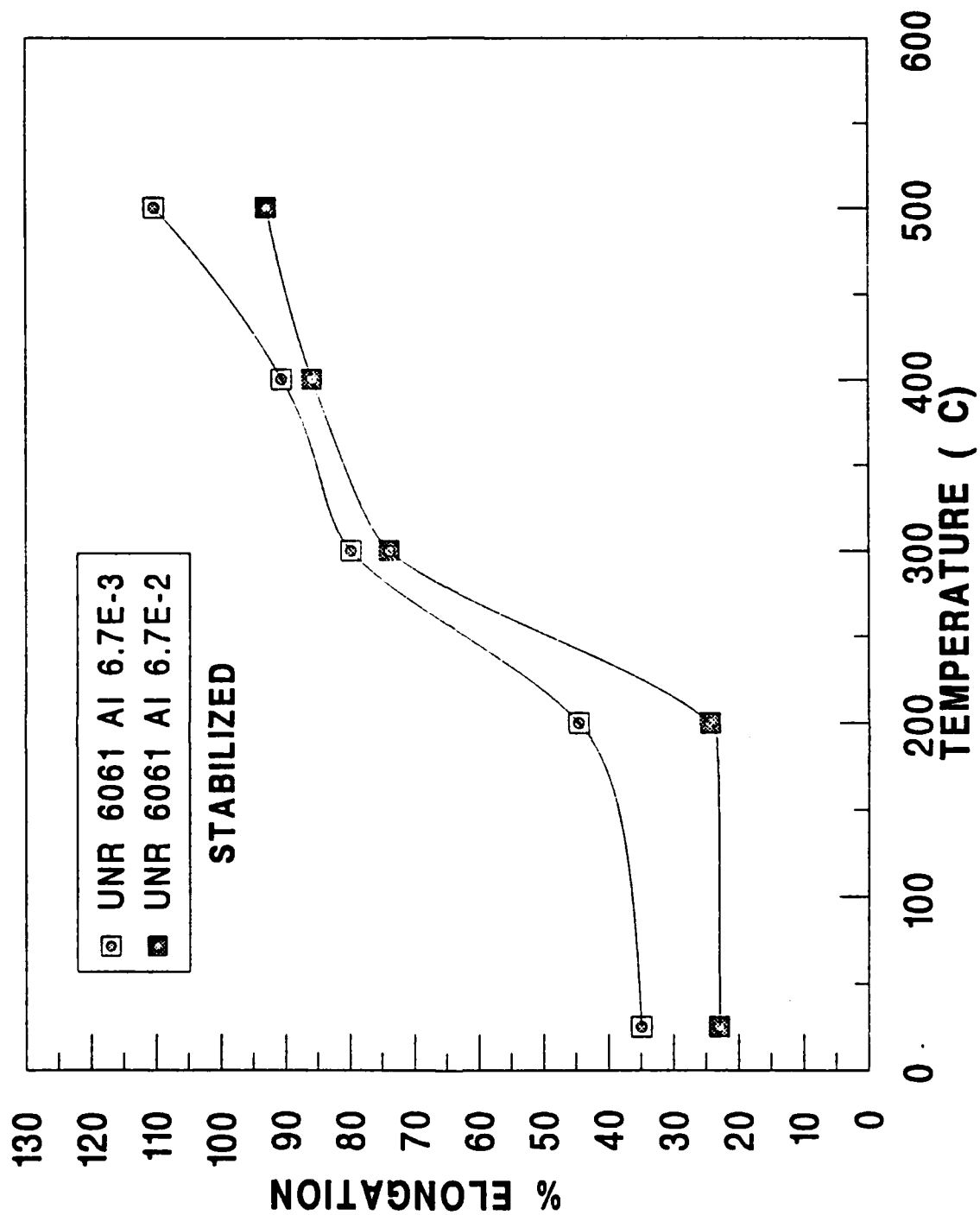


Figure 14. Ductility vs. Temperature for a  
Stabilized Unreinforced 6061 Al rolled at 350°C

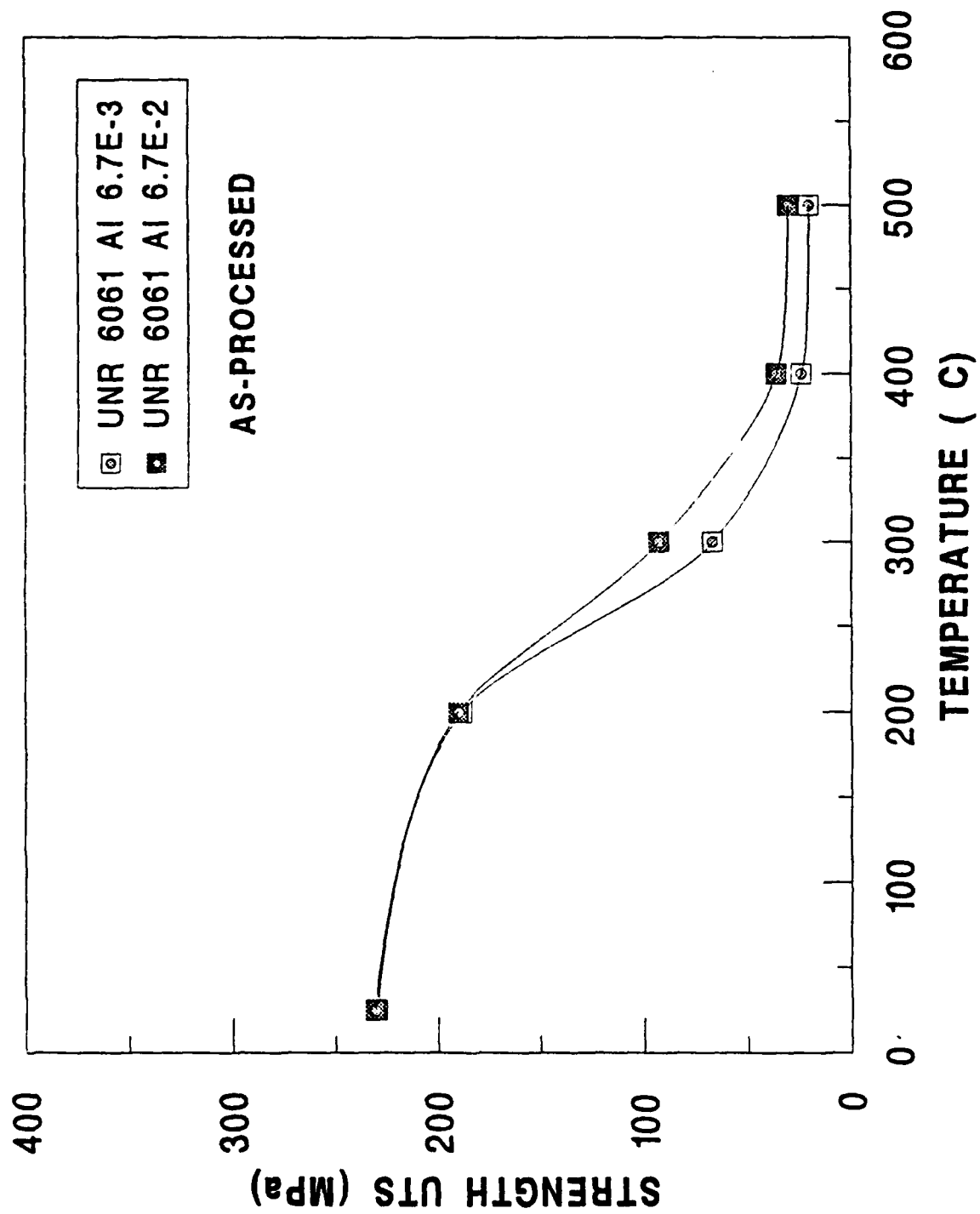


Figure 15. UTS vs. Temperature for an As-processed Unreinforced 6061 Al Rolled at 350°C

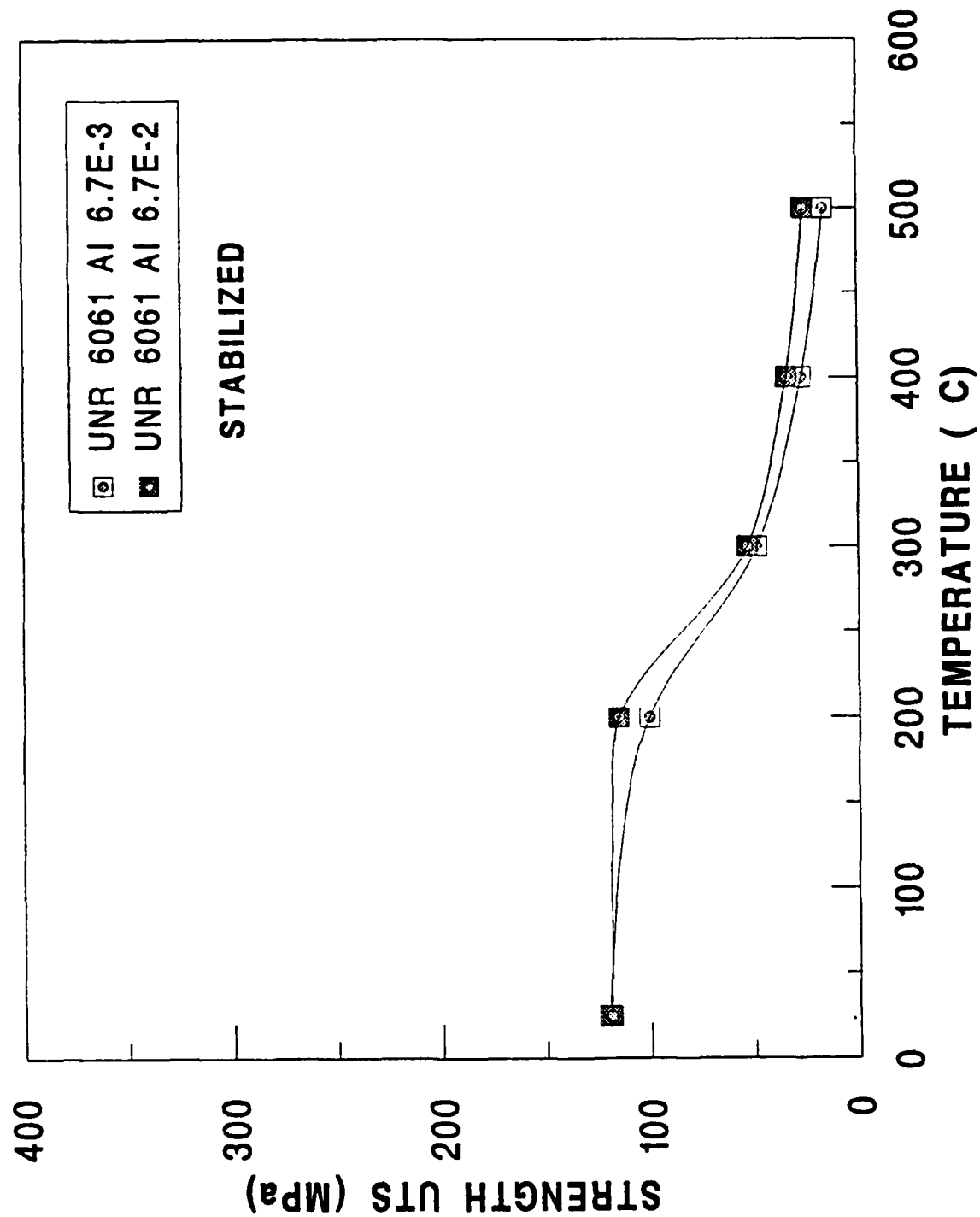


Figure 16. UTS vs. Temperature for a Stabilized Unreinforced 6061 Al Rolled at 350°C

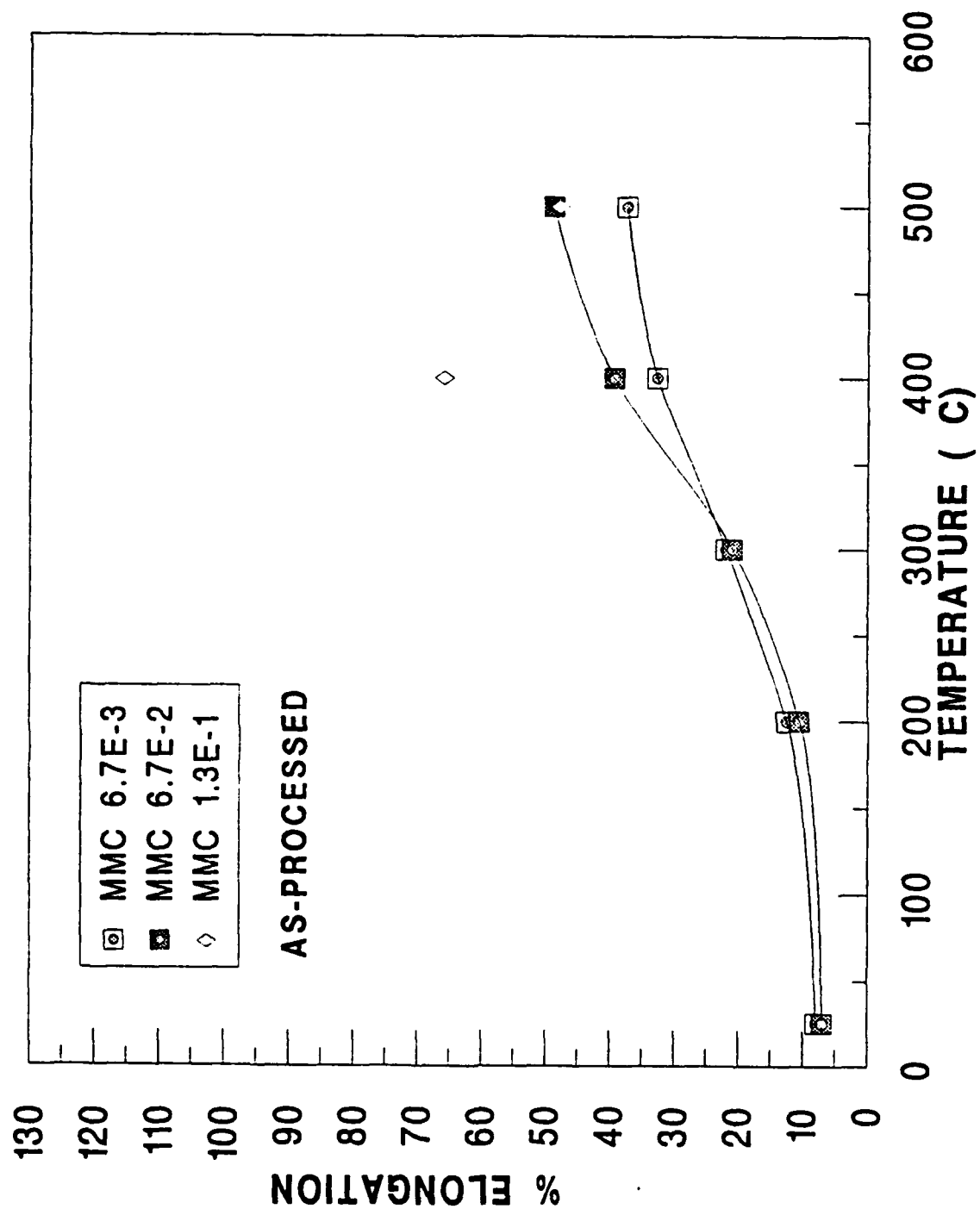


Figure 17. Ductility vs. Temperature for an As-processed MMC Rolled at 500°C

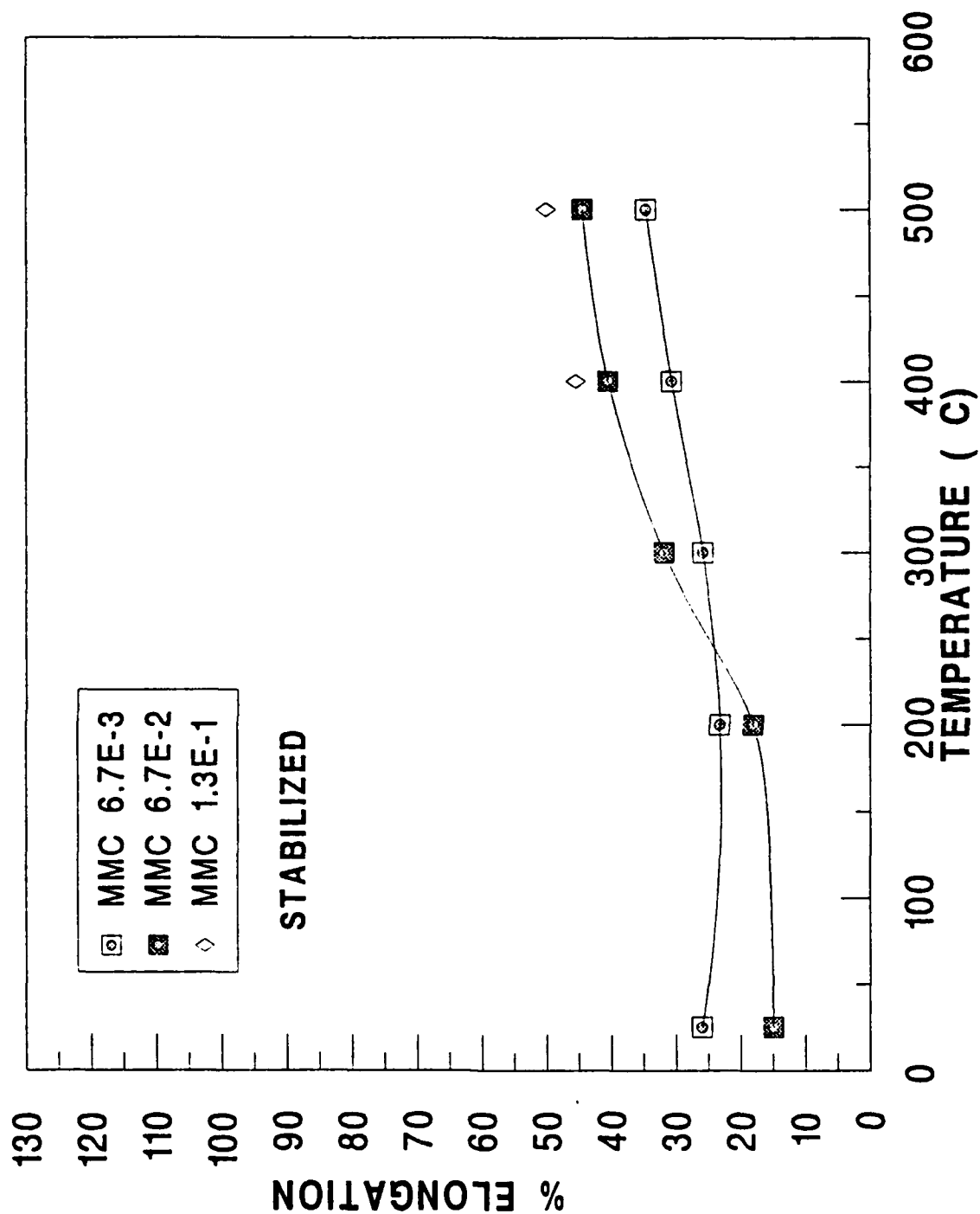


Figure 18. Ductility vs. Temperature for a Stabilized MMC Rolled at 500°C

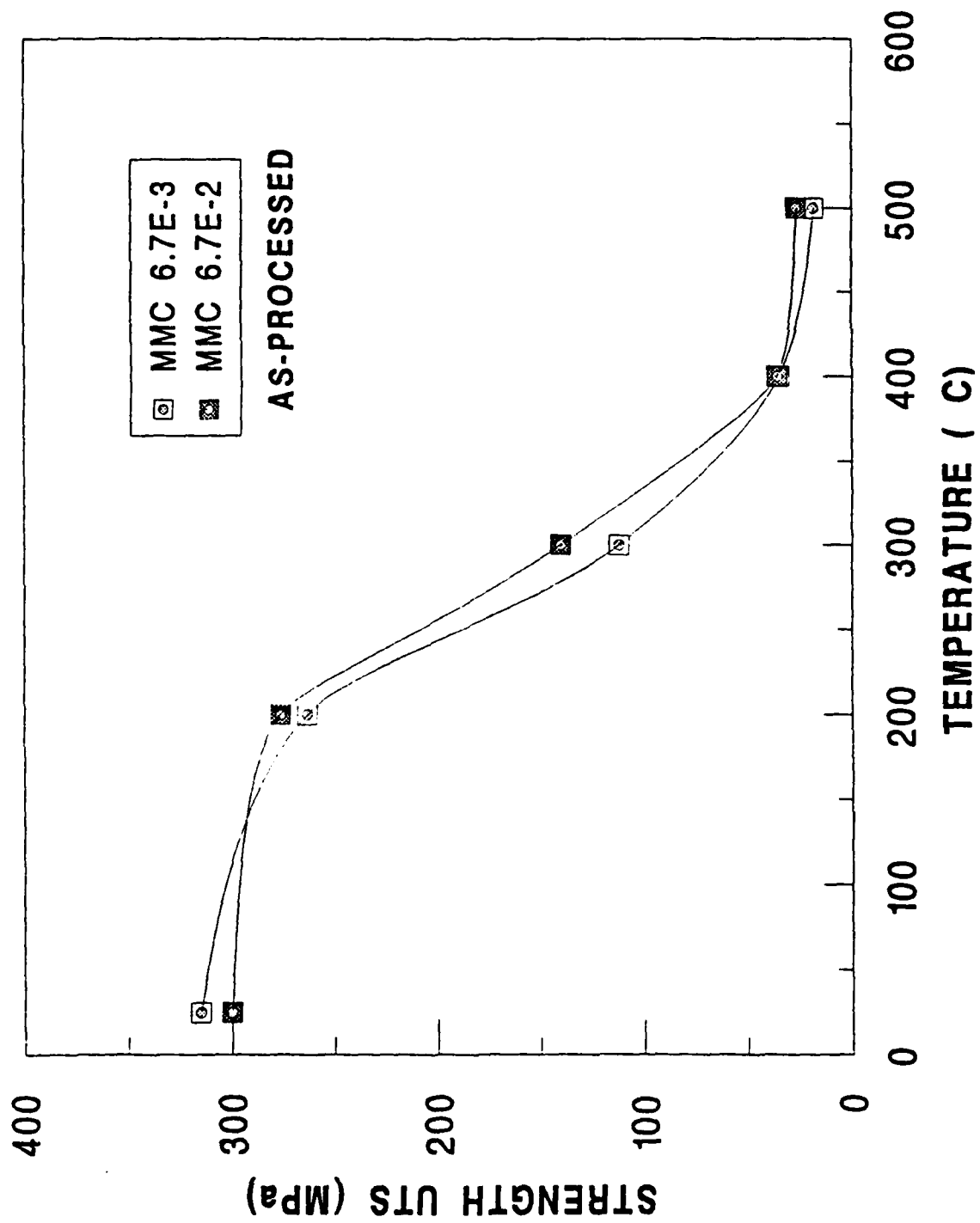


Figure 19. UTS vs. Temperature for an As-processed MMC Rolled at 500°C

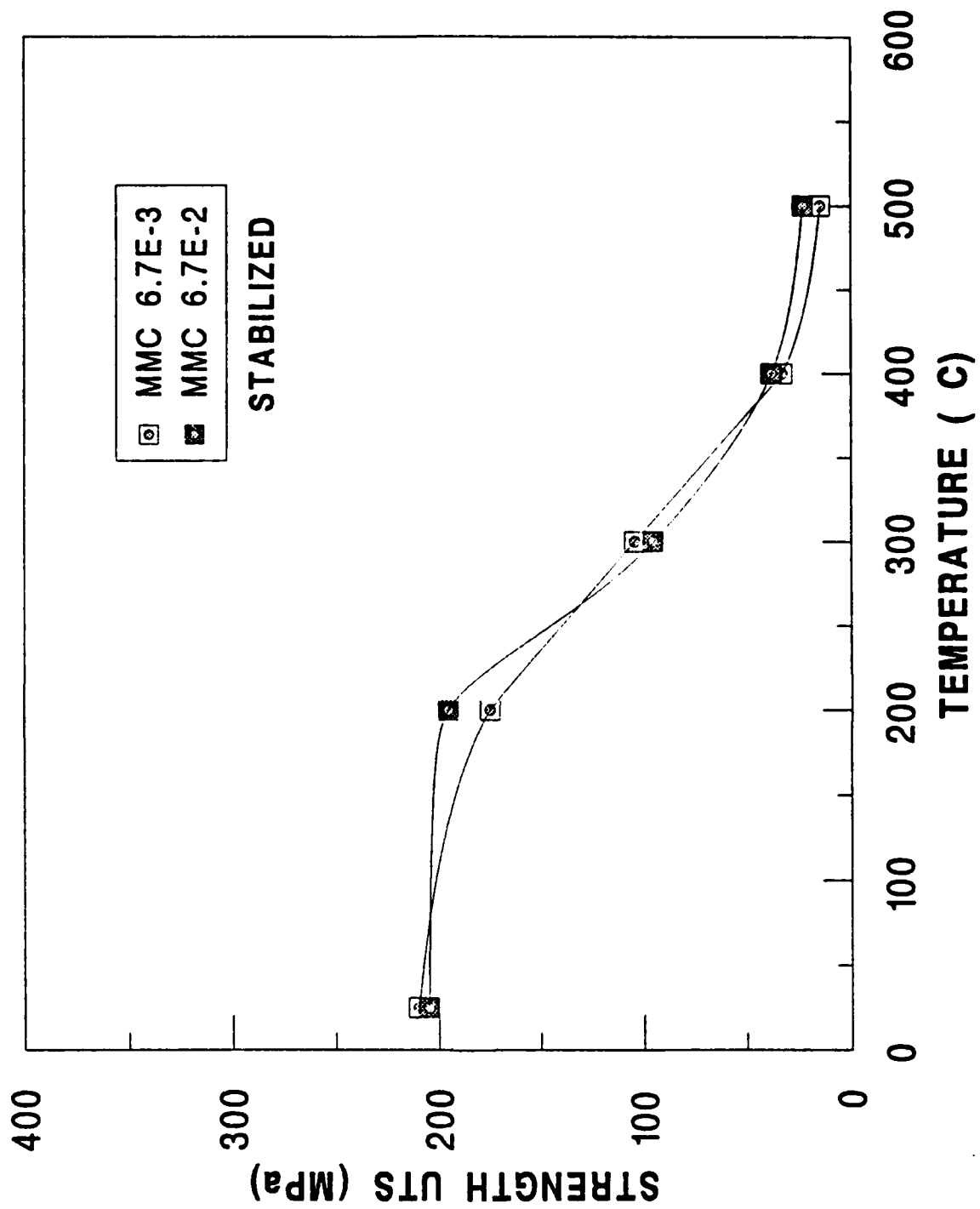


Figure 20. UTS vs. Temperature for a Stabilized MMC Rolled at 500°C



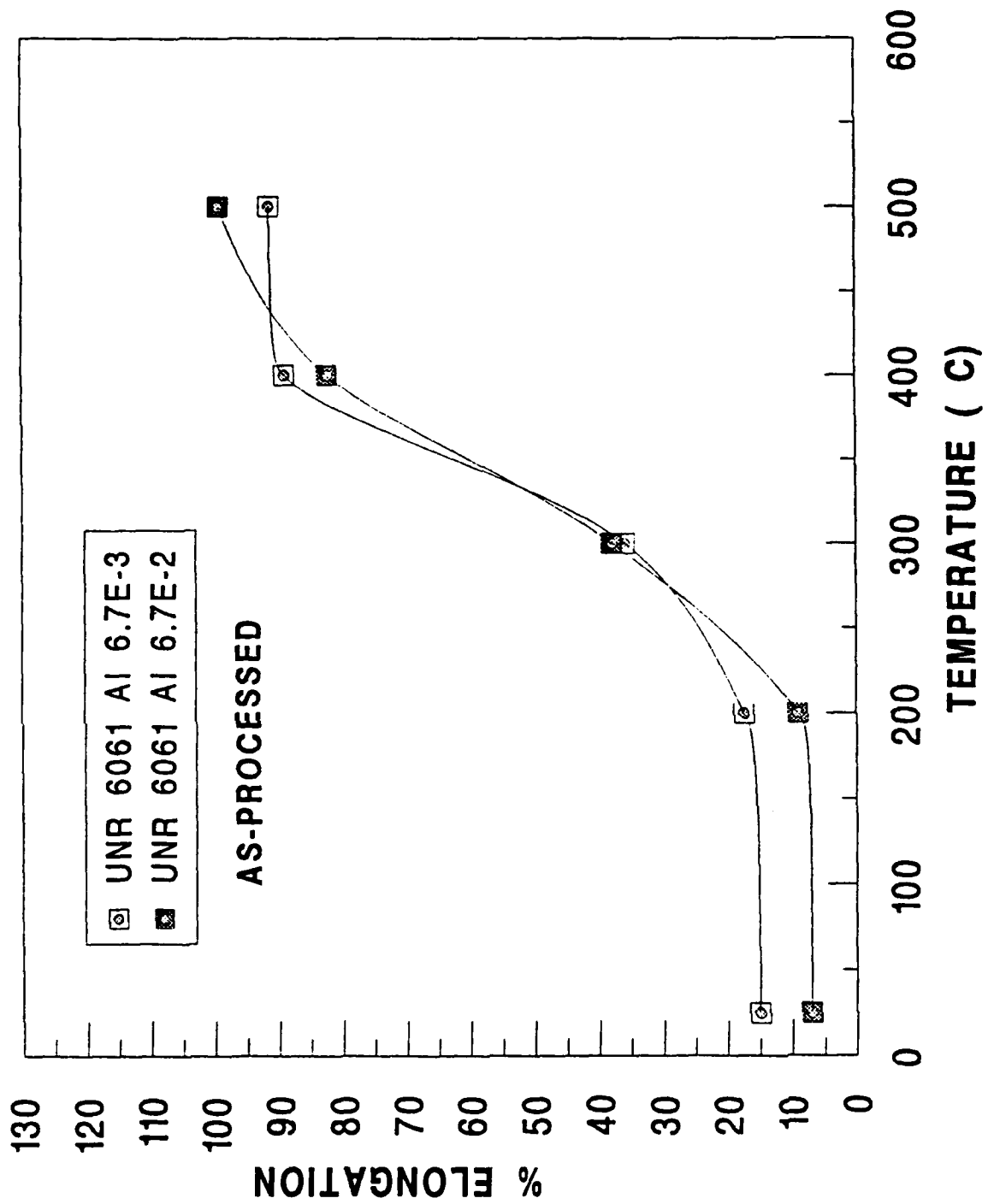


Figure 21. Ductility vs. Temperature for an As-processed Unreinforced 6061 Al Rolled at 500°C

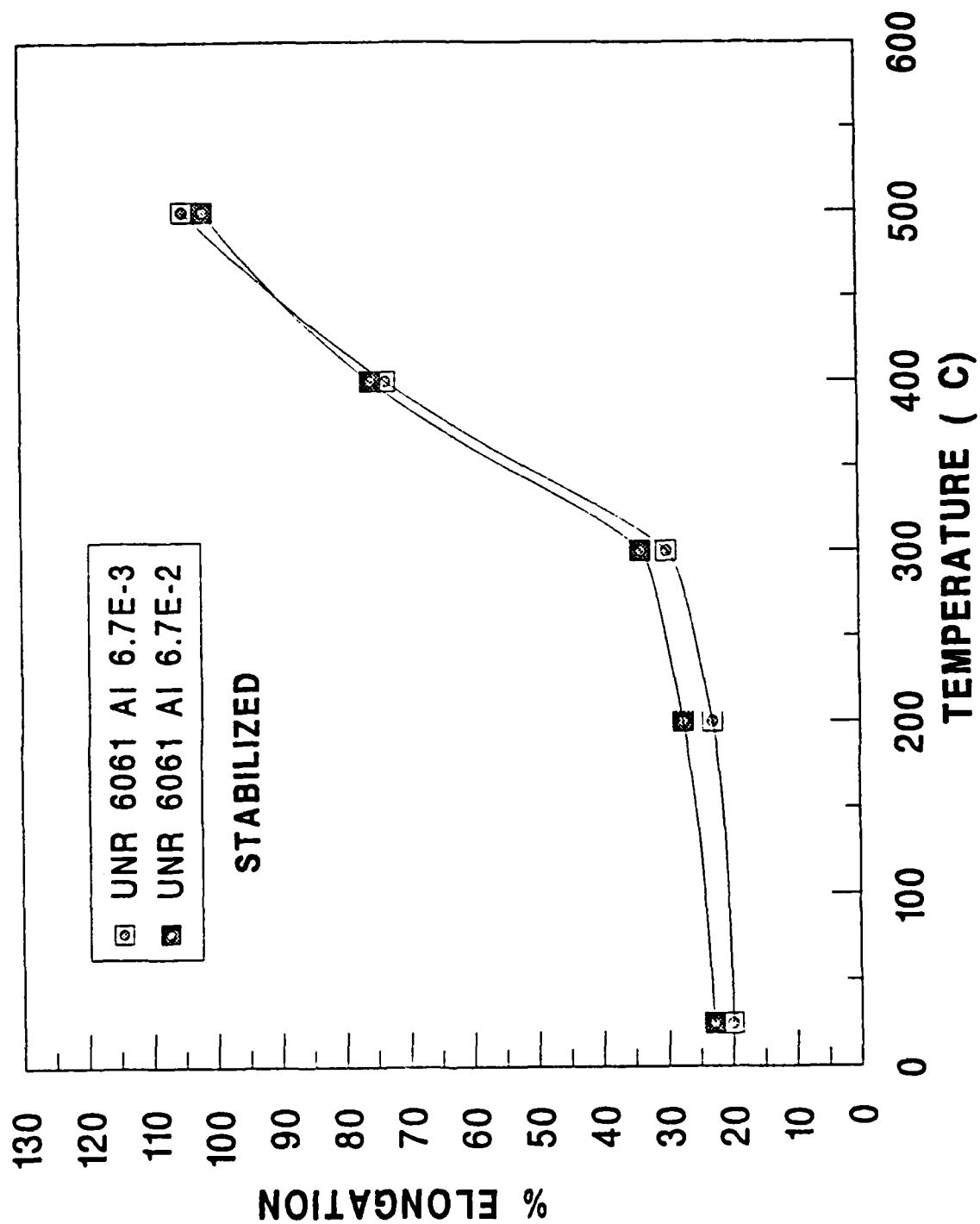


Figure 22. Ductility vs. Temperature for a Stabilized UNR 6061 Al Rolled at 500°C

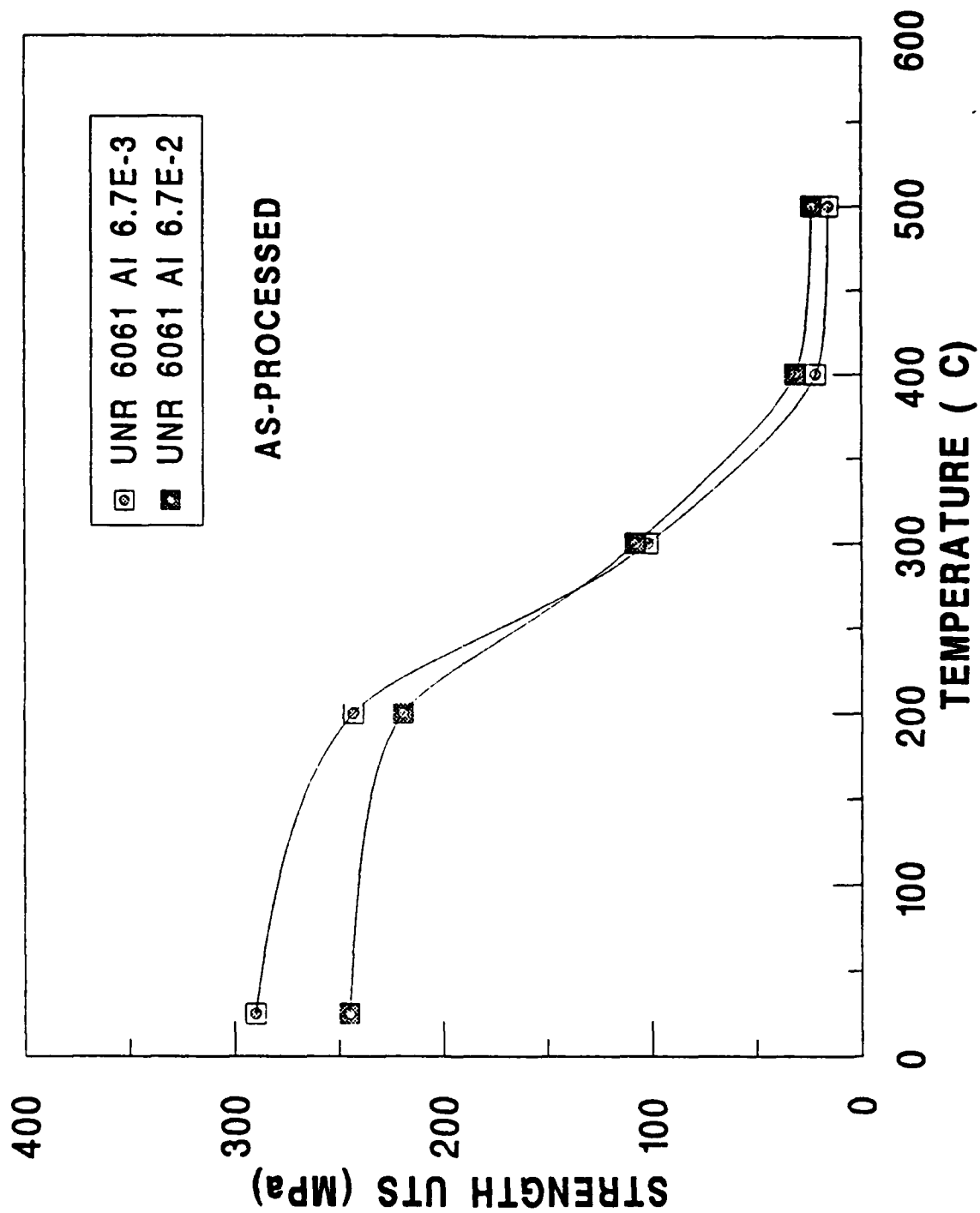


Figure 23. UTS vs. Temperature for an As-processed UNR 6061 Al Rolled at 500°C

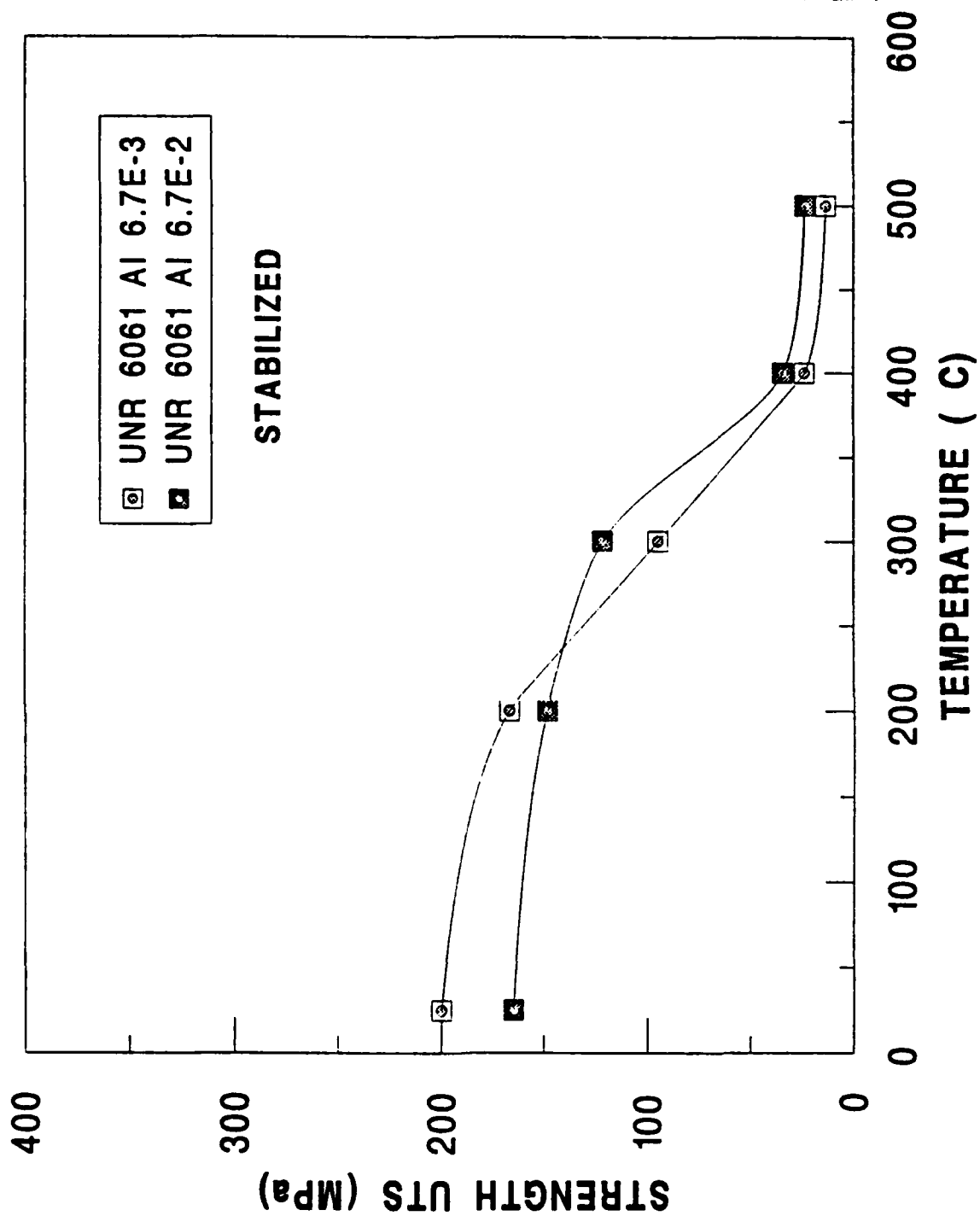
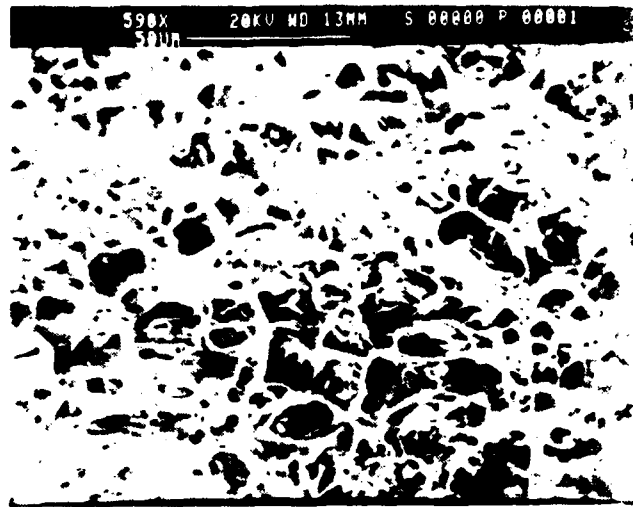
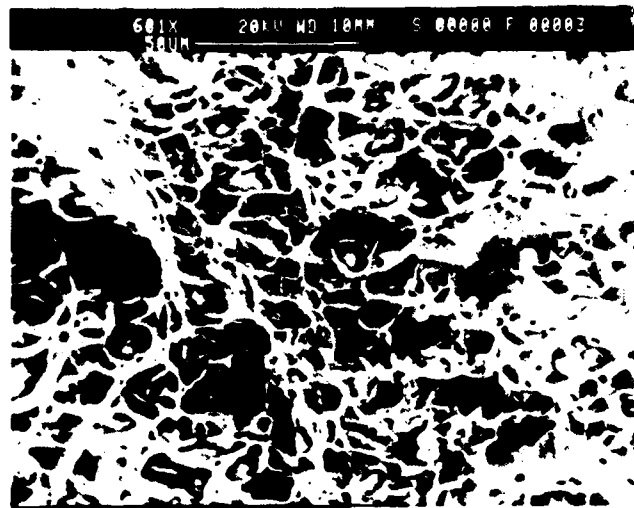


Figure 24. UTS vs. Temperature for a Stabilized UNR 6061 Al Rolled at 500°C

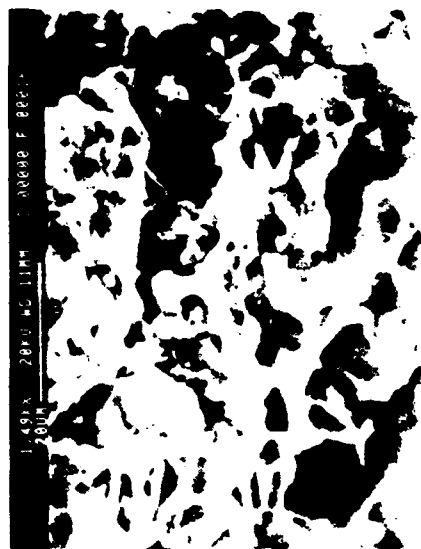


a. As-Processed MMC Rolled at 350°C, Tested at 200°C

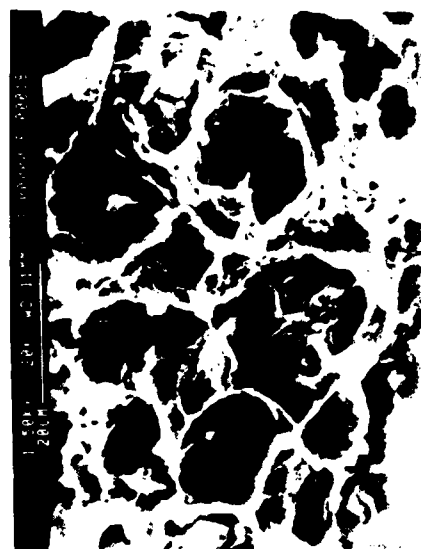


b. As-Processed MMC Rolled at 500°C, Tested at 200°C

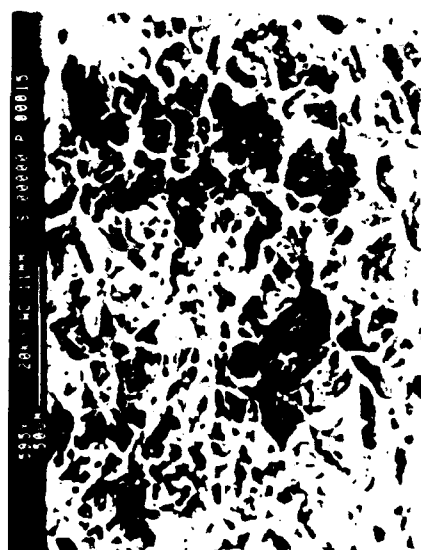
Figure 25. MMC Microvoid Formation and Coalescence



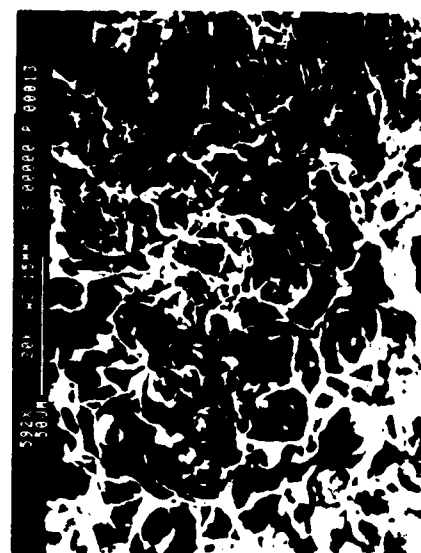
b. As Processed MMC Rolled at 350°C  
After 500°C Test (1.40 KX)



d. As-Processed MnFe Rolled at 500°C  
After 500°C Test (1,500 KN)

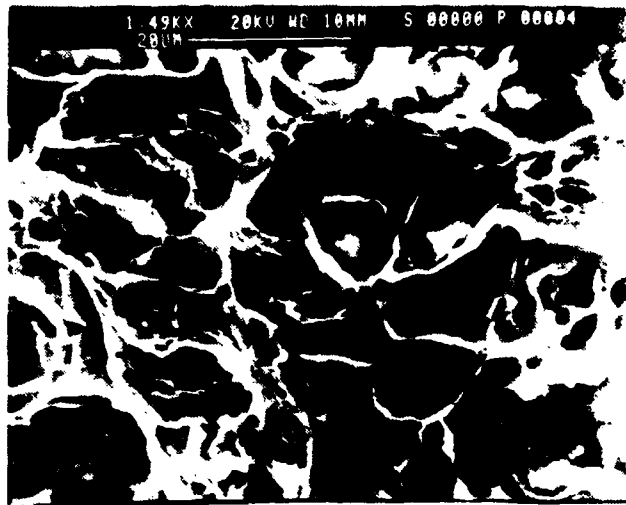


a. As-Processed MMC Rolled at 350°C  
After 500°C Test (595X)

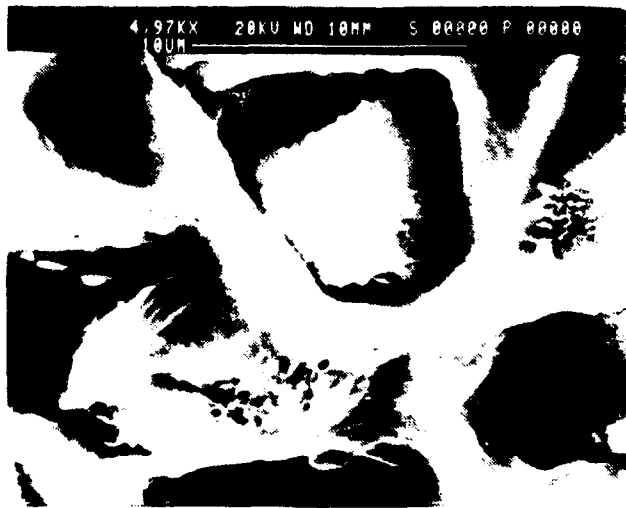


c. As-Processed MMC Rolled at 500°C  
After 500°C Test (502 X)

**Figure 26. Fracture Modes for MMCs Rolled at 350°C and 500°C After 500°C Test**



a. As-Processed MMC Rolled at 500°C After 200°C Test (1.49 KX)



b. As-Processed MMC Rolled at 500°C After 200°C Test (4.97 KX)

Figure 27. MMC Particles Shown Residing in Microvoids

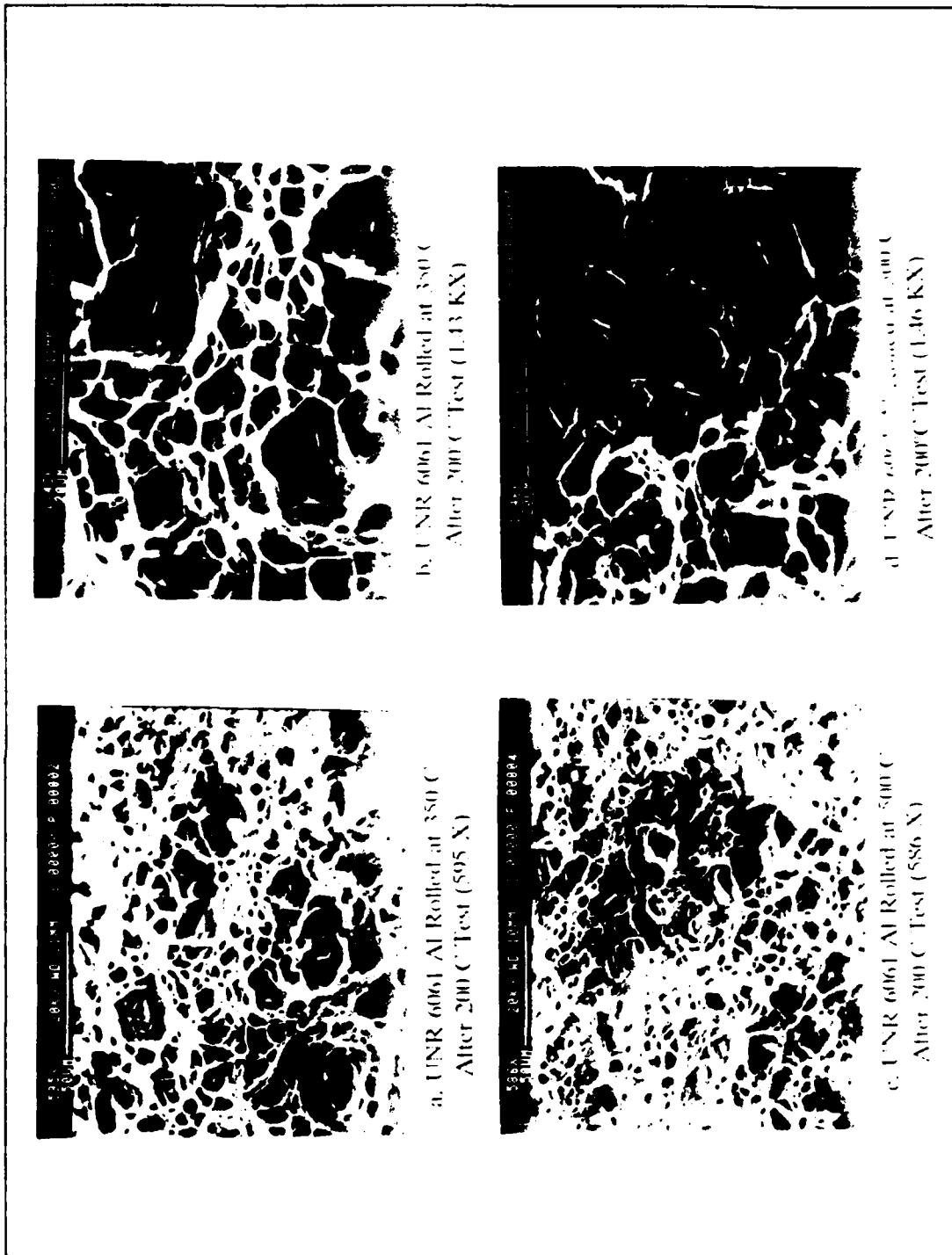


Figure 28. Unreinforced 6061 Al Fracture Modes for Materials Rolled at 350°C and 500°C



## V. DISCUSSION

The strength and ductility of MMCs and unreinforced 6061 Al is effected by thermomechanical processing. Several other factors effecting the mechanical response of these materials were analyzed, such as processing temperature, effects of stabilization anneals, homogeneity of particle distribution, solution treatment, and strain effects. The materials processed at 500°C exhibited higher strength when compared to those processed at 350°C for lower deformation temperatures.

The matrix strength was described earlier by the equation:

$$\tau_y = \tau_p + \tau_{\perp} + \tau_s + \tau_{ppt} + \tau_{gs}$$

For materials TMP at 350°C the  $\tau_{\perp}$  term is expected to be larger due to greater dislocation densities being generated at this lower processing temperature. Therefore, a higher strength should result. However, this study has shown 500°C TMP materials consistently display a higher UTS for testing temperatures up to 400°C. This is an indication that the precipitate strength ( $\tau_{ppt}$ ) and solute strength ( $\tau_s$ ) may contribute significantly to the MMC and unreinforced 6061 Al strength. It is believed that the 500°C TMP material contains a higher solute content due to it being in closer proximity to the solvus temperature. The amount of  $Mg_2Si$  precipitates will probably be less for the MMC processed at 500°C. However, the  $Mg_2Si$  precipitates for both 350°C and 500°C TMPs are expected to be coarser and widely spaced due to the

prolonged heating during rolling are therefore not likely to contribute significantly to the overall strength of the material. The strength resulting from dislocations and grain size do contribute, but the solid solution strengthening due to the higher solute content is apparently the major factor in the MMC's strength.

The MMC ductility was comparable to that of the unreinforced 6061 Al, especially after being stabilized. The effect of the stabilization was most apparent at lower test temperatures where it increased ductility and decreased the materials strength. Again, previous studies at the NPS have shown that the MMC matrix grain size was refined via PSN or recrystallization during 30 minute intervals of annealing between rolling passes. As a result of repeated PSN during successive cycles of deformation and annealing, more than one grain was nucleated per particle. Thus, matrix grain size was reduced increasing the material ductility [Ref. 14].

The TMP employed for this study greatly contributed to the homogeneous particle distribution, also increasing ductility. The TMP used two different rolling temperatures and a constant strain between passes. The alumina particles are relatively large, hard and non-deformable. These particles form clusters and show banding as a result of the extrusion process. The rolling deformation of the microstructure with an initially inhomogeneous particle distribution induces a strain which results in microstructural homogeneity. Rolling generates a high dislocation density in the vicinity of the particle clusters. Local strain hardening of the matrix forms resulting with increased strength [Ref. 15]. Dislocation density is lower at

locations further from the clusters where the material is weaker. As deformation proceeds, these weaker areas will deform more readily in comparison to the stronger regions near particle clusters. This results in redistribution of the clusters in the microstructure and leads to more uniform particle distribution [Ref. 16].

The stabilization process was most apparent at lower testing temperatures. This process increases ductility at higher temperatures and decreases UTS at these lower deformation temperatures. Ductility increases and lower strength are attributable to recovery and recrystallization. As mentioned earlier, the lower 350°C rolling process results in higher dislocation densities during the straining. Thus, the misorientation of boundaries evolved during annealing are likely greater within the deformation zones around the alumina particles [Ref. 16]. Materials rolled at the lower 350°C temperature exhibit a peak ductility of 75% at 400°C, reflecting finer, more highly misoriented grains, but then a decline in ductility at higher temperatures due to grain growth. The MMCs rolled at 500°C reach a peak ductility of 55% at approximately 400°C, but they maintain this ductility at higher temperatures suggesting a coarser but more stable grain size.

The effect of ceramic particles on the deformation is expected to alter as the temperature increases. At low temperatures, dislocations accumulate (pile-up) at the particles during deformation, and this can provide a large driving force for PSN of recrystallization on subsequent annealing. However, during deformation at elevated temperatures, dislocations are able to climb around the particles, thus increasing

ductility. With dislocation climb occurring, no deformation zones will be formed, and no PSN of recrystallization will occur on subsequent annealing. This is also expected to be true for the unreinforced 6061 Al [Ref. 17].

The results obtained also revealed that the solution treatment process prior to rolling did not effect the mechanical response of the MMC materials. The MMC material was extruded by Duralcan at sufficiently elevated temperatures to provide an essentially solution treated condition. Therefore, subsequent solution treatment did not effect the strength or ductility of the MMC.

The processing strain induced with an additional rolling pass from  $\epsilon_R = 2.2$  vs.  $\epsilon_R = 3.2$  also does not have a significant effect on ductility. Further studies are recommended to investigate the minimum strain during rolling required to maximize the materials mechanical response.

The SEM fractographs coupled with the ductility vs. temperature and UTS vs. temperature plots have shown that the MMC material behaves very much like the unreinforced 6061 Al. The MMC's enhanced ductility and strength, as well as its high modulus and wear resistance, improve its potential use for manufacturing processes.

## VI. CONCLUSIONS

### A. THERMOMECHANICAL PROCESSING (TMP)

The additional strain imposed on the extruded MMC enhances homogeneity of the  $\text{Al}_2\text{O}_3$  particle distribution.

### B. STRENGTH

1. TMP resulted in both the unreinforced 6061 Al and the  $\text{Al}_2\text{O}_3$  MMC samples having comparable UTS strengths.

2. The 500°C 6061 Al- $\text{Al}_2\text{O}_3$  MMC as-processed material attained the highest strength values.

### C. DUCTILITY

1. The processing strain induced with an additional rolling pass from  $\epsilon_R = 2.2$  vs.  $\epsilon_R = 3.2$  does not have a significant effect on ductility.

2. The solution treated samples and non-solution treated samples were compared and it was determined that the solution treatment process did not effect ductility.

3. At lower tensile testing temperatures (200°C/300°C) the stabilized materials had higher ductilities. Above 300°C testing temperatures (400°C/500°C) the stabilized materials did not show any enhanced ductility when compared to the as-processed material.

4. 500°C Al<sub>2</sub>O<sub>3</sub> MMC: The as-processed and stabilized materials both showed that they maintain an increasing trend in ductility at higher testing temperatures. The faster strain rates resulted in higher ductility.

5. All plots show the TMP unreinforced 6061 Al is always similar or higher in ductility than the MMC for both of the 350°C and the 500°C TMP rolling sequences.

6. 350°C vs. 500°C MMC: The 350°C MMC is higher in ductility up to 400°C testing temperature after which its elongation drops significantly. The 500°C MMC does not achieve as high a ductility, but maintains its ductility from 400°C to 500°C testing temperature.

#### **D. SEM/FRACTOGRAPHY**

1. At lower test temperatures the MMC and unreinforced 6061 Al display similar fracture modes, microvoid formation and coalescence.

## **VII. RECOMMENDATIONS FOR FURTHER STUDY**

1. Conduct transmission electron microscopy (TEM) and assess grain/subgrain structures resulting from varying rolling passes. Analyze grain sizes of microstructures after being mechanically tested at the various temperatures.
2. Investigate effects of processing upon the unreinforced matrix 6061 Al using TEM.
3. Determine effects of TMP upon fatigue and fracture characteristics.
4. Test and analyze MMC materials with a smaller particle size.
5. Investigate the minimum number of rolling passes required to maximize the MMC mechanical response.

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