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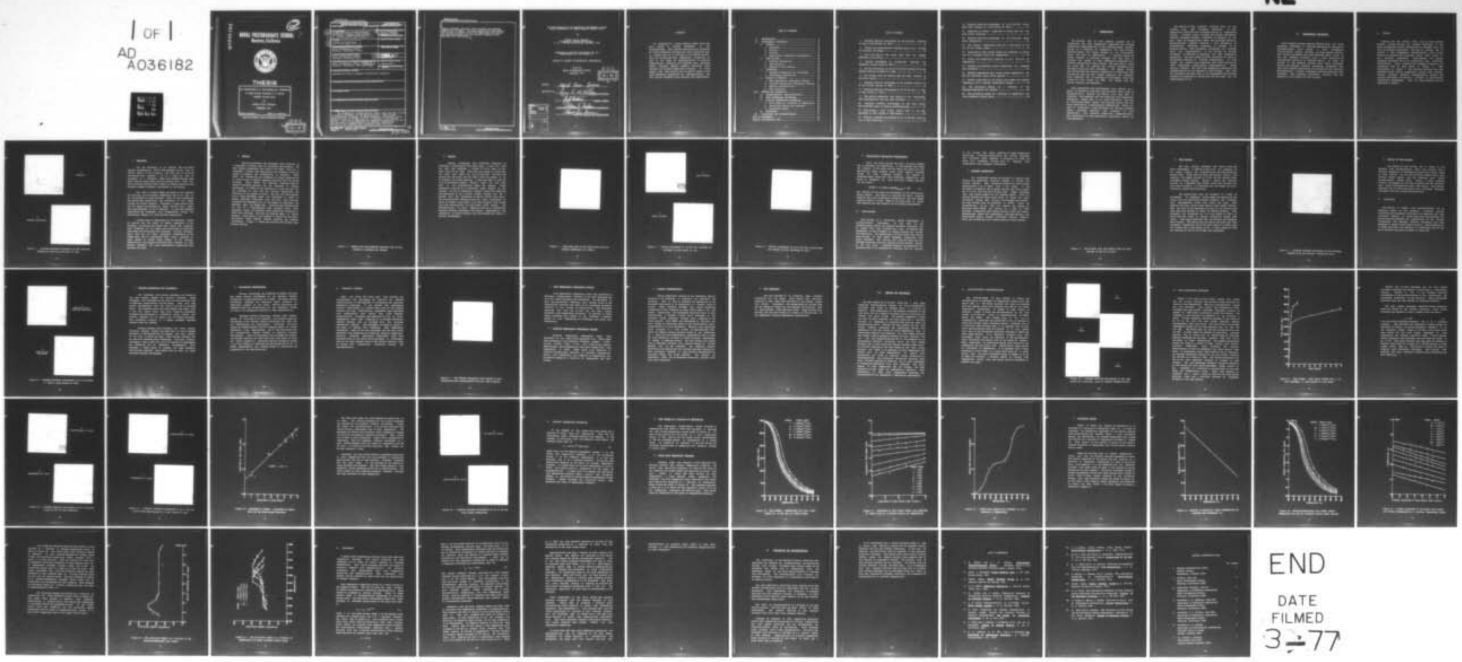

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NAVAL POSTGRADUATE SCHOOL

Monterey, California

THESIS

AN INVESTIGATION OF THE MECHANICAL PROPERTIES
OF WARM ROLLED ALUMINUM-17.5 WEIGHT
PERCENT COPPER ALLOY

by

ALFRED LOUIS CIPRIANI

DECEMBER 1976

Thesis Advisor: Terry R. McNelley

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An aluminum-17.5 weight percent copper alloy was warm rolled to achieve refinement of the microstructure. This refined microstructure consisted of finely dispersed intermetallic Al_2Cu particles with an average size of 1.2 microns in an aluminum matrix. This led to improved room temperature properties as well as the onset of superplasticity at elevated temperatures. Ductility and toughness were increased almost six-fold,

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AN INVESTIGATION OF THE MECHANICAL PROPERTIES OF WARM ROLLED ALUMINUM-17.5 WEIGHT PERCENT COPPER ALLOY

by

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Lieutenant, United States Navy
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Submitted in partial fulfillment of the requirements for the degree of

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December 1976

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ABSTRACT

An aluminum-17.5 weight percent copper alloy was warm rolled to achieve refinement of the microstructure. This refined microstructure consisted of finely dispersed intermetallic Al_2Cu particles with an average size of 1.2 microns in an aluminum matrix. This led to improved room temperature properties as well as the onset of superplasticity at elevated temperatures. Ductility and toughness were increased almost six-fold, despite slightly decreased yield strength and maximum compressive strength. It would appear that warm working can be used to enhance both room temperature properties and superplasticity, and that further research could maximize this improvement.

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I. INTRODUCTION

The original goal of this research program was to investigate the room temperature properties and elevated temperature superplasticity of the aluminum-copper eutectic system as a function of composition and warm rolling. Aluminum-copper was chosen because of its non-reactive nature and ease of fabrication. A eutectic system was chosen because no previous attempts had been made to evaluate the change in room temperature properties of a eutectic system due to warm rolling. The goal stated above was never met, as casting and rolling procedures took extensive trial and error refinement. The copper content was reduced from the eutectic composition to facilitate warm rolling and only one warm worked condition was produced. This condition was an aluminum-17.5wt.%copper alloy warm rolled to a true strain of 150%. The ground work for further research was performed.

Many researchers have investigated warm rolling as a means to produce an ultrafine microstructure which leads to superplasticity. However, few efforts have been made to correlate this research with the resulting room temperature properties. Sherby (Ref. 1) is a major exception. His work on ultra-high-carbon steels has produced results that are being described as a metallurgical breakthrough. There is a need to investigate other materials for the same tendency toward increased ductility and toughness at room temperature, with elevated temperature superplasticity. This correlation of room temperature properties cannot be overemphasised.

The results of this research, although short of the original goal, were extremely promising. At room temperature, ductility was increased greatly in compression testing. Although yield strength and maximum compressive strength decreased slightly, toughness, as measured by the area under the stress-strain curve, was increased six-fold. Microstructural analysis revealed a fine dispersion of intermetallic particles, averaging 1.2 microns, in an aluminum matrix. This refinement eliminated the cracking which was characteristic of the failure of as-cast specimens. In addition to improving the room temperature properties, the warm worked material exhibited the onset of superplastic behavior at elevated temperatures. In this region, the results were compatible with those reported by other researchers investigating superplasticity. These results indicate that both room temperature properties and superplasticity can be enhanced by warm rolling. Now that the groundwork has been laid, it is recommended that further research be performed to extend both the data base and the understanding of it.

II. EXPERIMENTAL PROCEDURES

This research program required tedious trial and error procedure refinement to develope reproducible castings of aluminum-copper alloy having a fine homogeneous microstructure without porosity or other casting defects. Warm rolling procedures were then developed which broke up the cast lamellar structure into a fine dispersion of intermetallic grains in an aluminum matrix. As these two aspects of the research program were being developed, optical and scanning electron microscope procedures were used which enabled observation and analysis of the resultant microstructure. Finally, mechanical testing procedures were developed that produced consistent, accurate data for analysis of the effects of warm working.

A. CASTING

Casting was the crucial first step in this research program. Limited melting and casting facilities at the Naval Postgraduate School necessitated an extended trial and error procedural development. The goal in casting was to produce a fine homogeneous microstructure, free of such defects as porosity and inclusions which would result in poor material properties. Additionally, the casting procedure developed had to be reproducible and, therefore, controllable. Early castings were plagued by porosity and inclusions such as those shown in figure 1. Both micrographs in figure 1 show fracture surfaces from cast materials which failed prematurely during warm rolling. Casting involved material considerations, melting and pouring procedures, and finally , a method of quantitative composition measurement. Each of these subjects will be described in detail.

A.
POROSITY

B.
ALUMINA INCLUSION

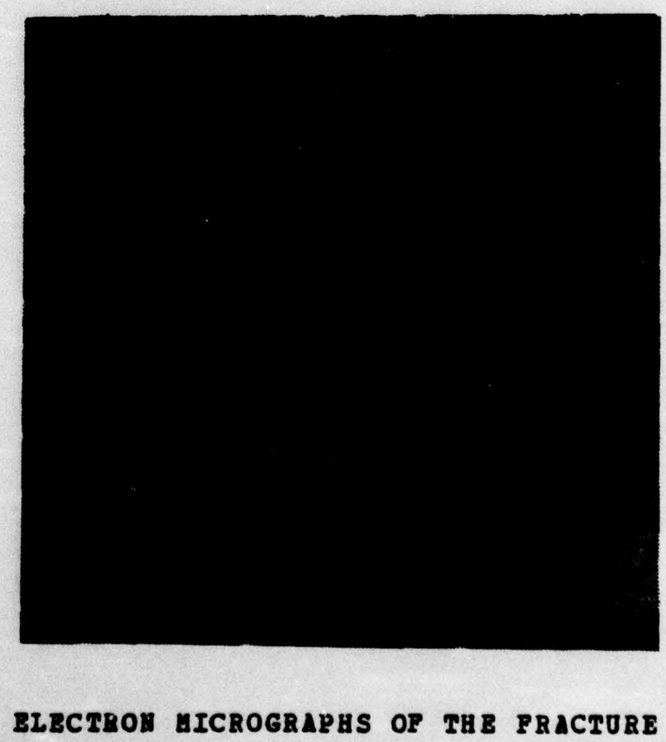


Figure 1 - SCANNING ELECTRON MICROGRAPHS OF THE FRACTURE SURFACES OF CAST Al-25.5WT.%Cu AT 700X.

1. <u>Materials</u>

The raw materials to be alloyed were the first casting consideration. Stock 1100 aluminum in the form of rolled sheets 22 cm in length, 2 cm wide, and .32 cm thick was sheared into squares 2 cm on a side for ease of placement in a crucible. These were sheared because less local melting would occur than by cutting with a band saw. This localized melting would result in alumina formation and thereby increase resultant inclusions in the casting.

The form in which copper was added to the aluminum was critical, as the furnace temperature was to be kept as low as possible to avoid porosity. Thus the only mechanisms for mixing were dissolution and diffusion of the copper through the molten aluminum. Therefore, a high surface area for the copper was provided by using (99.9% pure) copper shot approximately .15 cm in diameter. In this way adequate dissolution and diffusion were accomplished without the detrimental effects of a high melting temperature.

A final point of importance in the materials aspect of melting was the order of adding components to the crucible. By placing the copper shot below the aluminum, less oxidation of the copper and elimination of resultant cold shuts and inclusions were achieved. In early castings, where this was not done, incomplete dissolution of the copper shot, due to a protective oxide cover, resulted in whole pieces of copper being present in the casting. Additionally, the alloying materials were cleaned to keep surface oxidation and other environmental impurities from entering the melt.

2. Melting

Melting procedures were developed which resulted in a homogeneous casting, free of inclusions and porosity. The best melting temperature was found to be 978°K. This was a compromise between high dissolution and diffusion rates for copper in aluminum and minimization of porosity. Below 923°K the dissolution and diffusion of copper required ten to twelve hours to achieve adequate alloying with the molten aluminum. Above 1023°K the resultant castings began to exhibit extensive porosity and the formation of alumina inclusions. At 978°K it was necessary to allow one hour for the aluminum to melt and for initial dissolution and diffusion of the copper to occur. The melt was then stirred with a clean stainless steel rod preheated to 423°K to reduce moisture carry-over into the melt. This stirring increased the diffusion rate of the copper by improving the gradient near the copper shot. Stirring also necessitated the use of a clay-graphite type crucible since other crucibles would not maintain their structural integrity at 978°K with stirring. The melt was stirred again in thirty minutes, after which it was allowed to settle for one and a half hours, which further reduced porosity and alumina inclusions. Figure 2 shows the crucible and furnace used during melting.

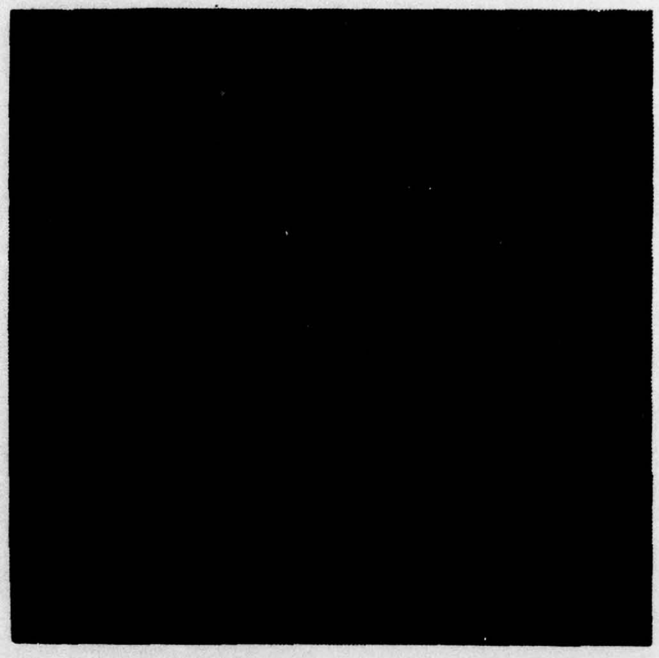


Figure 2 - FURNACE AND CLAY-GRAPHITE CRUCIBLE USED IN THE MELTING OF ALUMINUM AND COPPER.

3. Pouring

Pouring procedures were extremely important in achieving a controllable fine grain size. A cast iron mold was used that produced a billet 17.75 cm. long, 3.6 cm. wide, and 2.75 cm. deep. This mold was placed in water with the level just below the top of the mold. The melt was poured into the mold, and the water level was raised so as to submerge the entire casting. A hose was used to keep the mold surroundings flushed. Figure 3 shows the cast iron mold in the configuration ready for pouring. These procedures resulted in a controlled rapid cooling of the casting. By way of comparison, figure 4 shows the resulting microstructure of (A) a slowly cooled casting and (B) a droplet that was solidified in direct contact with water. The microstructure resulting from the above procedures is shown in figure 5 and proved to be only slightly more coarse than this uncontrolled, but very rapid, water quench. The microstructure, in fact, became so fine as to necessitate scanning electron microscopy for greater magnification and resolution of minute details that were beyond the limits of optical microscopes.

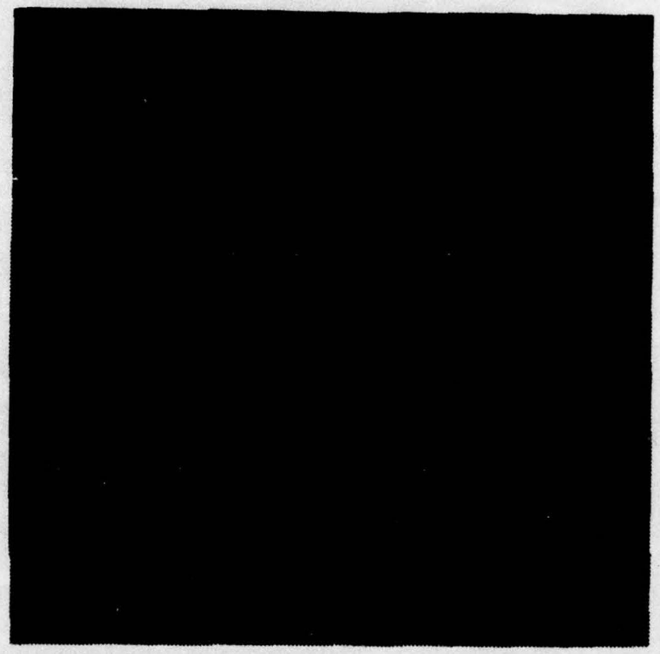
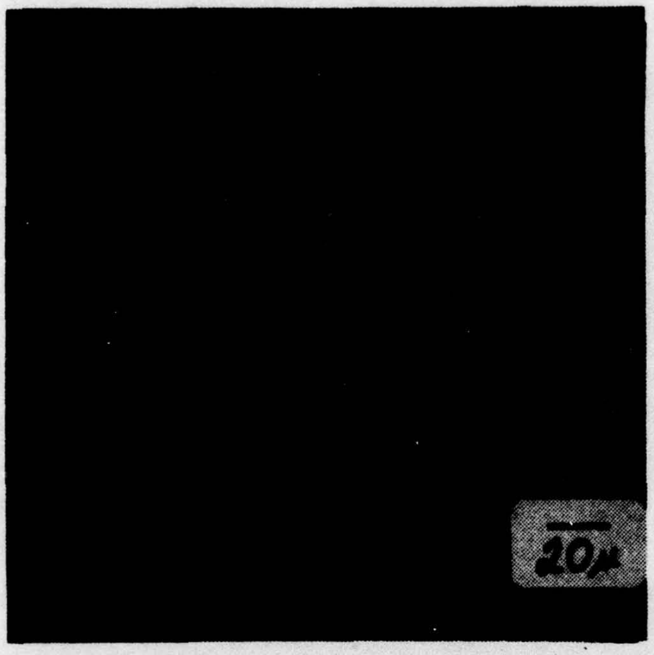


Figure 3 - CAST IRON MOLD IN THE WATER BATH USED FOR RAPIDLY QUENCHING AL-CU MELTS.



A.
SLOW COOLING

B.
RAPID COOLING

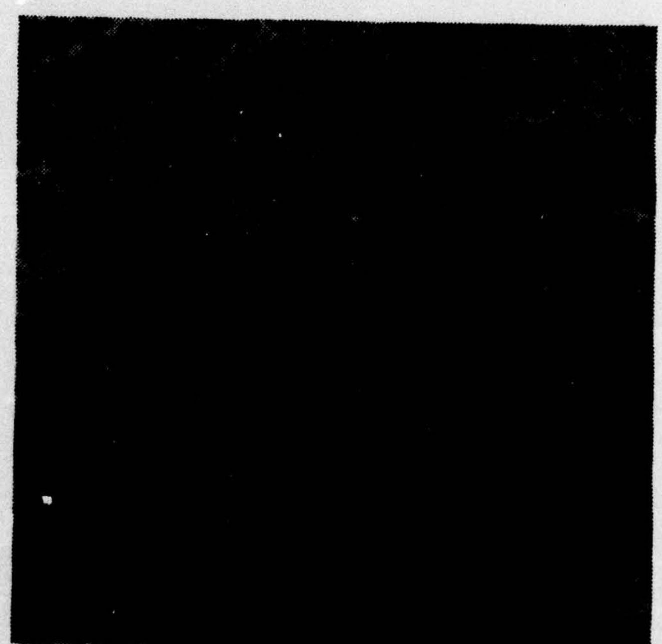


Figure 4 - OPTICAL MICROGRAPH OF AL-25Wt.%CU CASTINGS FOR DIFFERENT COOLING RATES AT 400X.

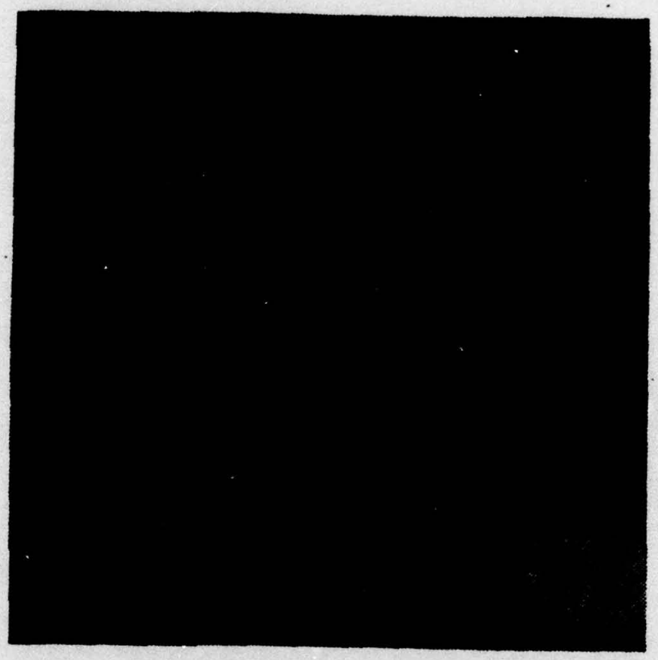


Figure 5 - OPTICAL MICROGRAPH OF AL-17.5Wt.%CU CASTING FROM THE ADOPTED CASTING PROCEDURE AT 800X.

4. Quantitative Composition Measurements

After the first casting was made, the major problem was to determine the composition. It was decided that using the density of the alloy and the lever rule along with the equilibrium phase diagram would yield results accurate enough for this research program. The density of aluminum-copper alloys for three different compositions was found in reference (2). From this data a simple equation was the result.

$$\text{Wt.\%Cu} = (1.2534 - 3.3208/\rho_{alloy}) \times 100 \qquad (1)$$

The density of the alloy was determined by the differential weight method . The results of applying the above equation compared well with microscopic observations and were deemed accurate within 1 wt.%Cu based on the data used to derive this equation and the accuracy of the density measurement.

B. WARM ROLLING

Warm rolling is a relatively recent development in microstructural refinement. In the case of the aluminum-copper system the lamellar eutectic structure can be broken into fine discrete particles. To achieve this refinement, it was necessary to closely control the as-cast microstructure, the temperature, and the strain rate during warm rolling. In this sense equipment limitations were quite profound. The rolling mill used for this work was designed primarily for cold rolling and had limited control of the roll bite. A separate furnace was installed, but specimens had to be transferred a distance from the furnace to the rolling mill which resulted in large temperature variations. A material preparation and rolling procedure were developed which resulted in strains up to 150%, and which were ultimately limited more by specimen size requirements than equipment limitations.

1. Material Preparation

The solidified casting was milled to a square cross section with sufficient material removed to exclude any surface defects. This operation resulted in three pieces approximately 5 cm in length with 2 cm. sides. It was necessary to separate the original casting into three pieces for it to fit into the warm rolling oven. These specimens were then finely sanded with 3/0 sanding wheels. Since any surface defects and sharp edges would be sites for crack initiation during warm rolling, this sanding was helpful. The warm rolling oven was preheated to 783°K. Since the intermetallic, Al_2Cu, does not exhibit any degree of ductility below 623°K, and because of a 75°K to 150°K specimen temperature drop during rolling, 783°K was chosen. Additionally, above that temperature, localized melting was experienced during furnace controller cycling. Figure 6 shows the rolling mill and furnace used for warm rolling.

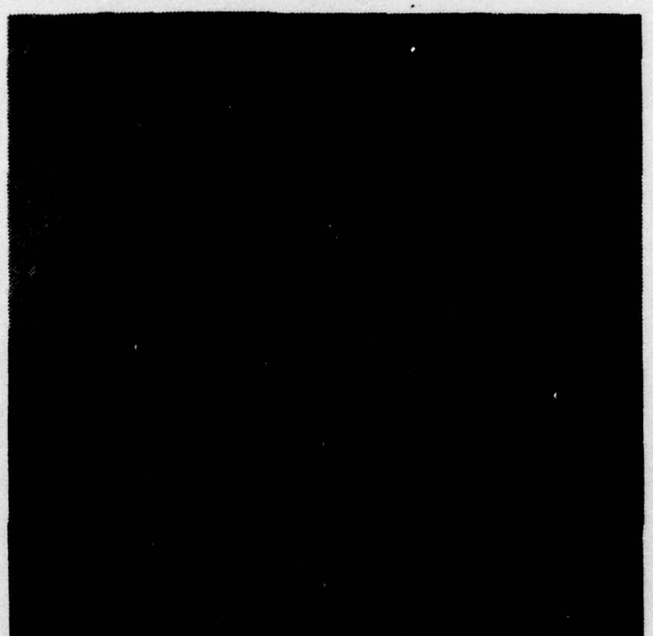


Figure 6 - THE ROLLING MILL AND FURNACE USED FOR WARM ROLLING OF THE AL-CU ALLOY.

2. Warm Rolling

The warm rolling procedure was also a trial and error development. A greater strain rate was possible as the copper content was lowered and as the microstructure became finer. These were the major motivating factors for reducing the copper content to 17.5% and for further grain refinement during casting. Figure 7 shows the fracture surface of a specimen that failed during warm rolling with less than 5% strain. It contained over 31% copper and had a relatively coarse microstructure.

The rolling mill could be adjusted in 0.0025 mm increments. By adjusting roller speed and separation the strain rate was maintained between 0.006/sec and 0.008/sec with approximately 0.75% true strain per pass. Each specimen was flop rolled three times at each roller setting. This was done to all specimens at eight to ten settings and then they were allowed to anneal for fifteen minutes. This procedure was continued up to a true strain of 100% , at which time the sound of rolling initiation became less sharp. This sound indicated that the material was becoming more ductile. The strain rate and strain per pass were then increased continually until the final pass at a strain rate of 0.05/sec and a true strain of 6%. A total strain of 150% was achieved on the Al-17.5wt.%Cu cast specimens.

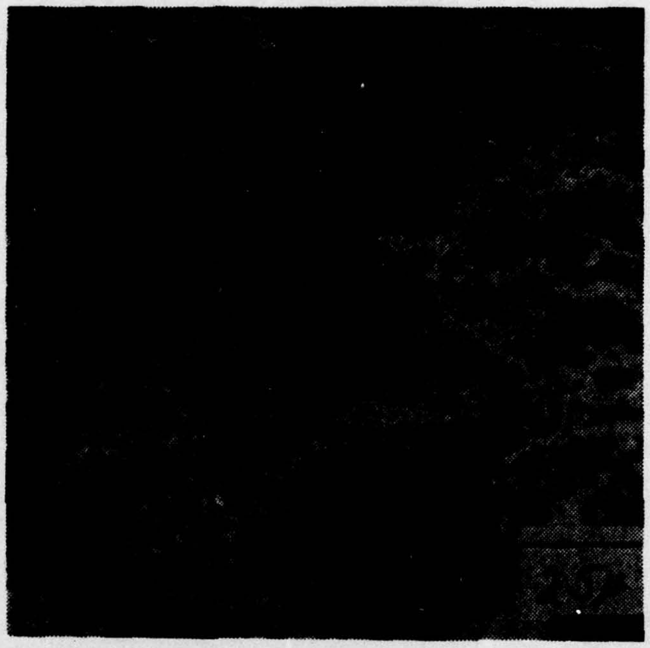


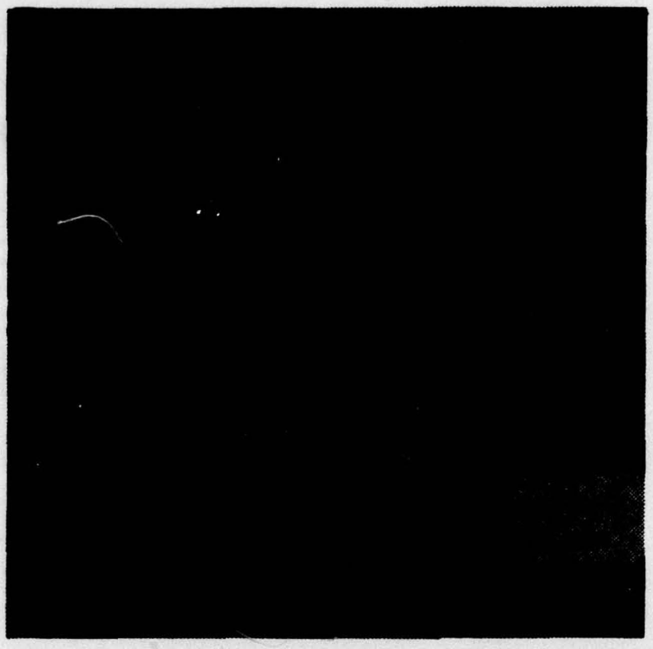
Figure 7 - SCANNING ELECTRON MICROGRAPH OF THE FRACTURE SURFACE OF AN AL-31Wt.%CU CASTING AT 650X.

3. Effects of Warm Rolling

The purpose of warm rolling was to break up the skeletal cast microstructure, which was brittle, into a discreet dispersion of intermetallic particles which would be more ductile. As the cast structure became finer, warm rolling became easier, and the resultant warm rolled microstructure became finer. Figure 8 shows scanning electron micrographs of the cast structure and the resultant warm rolled microstructure after 150% strain. Particularly important was the fine sub-micron size particles dispersed throughout the warm rolled microstructure.

C. MICROSCOPY

The ability to resolve fine microstructures was an important aspect of this research. Initially, optical microscopy was considered sufficient for observing the cast structure; however, as finer grain size was achieved, the microstructures exceeded the capabilities of the optical microscopes. The scanning electron microscope was then employed to resolve these minute details. Magnifications up to 8000X were used, for instance, to investigate some of the finer discreet particles as shown in figure 8B.



A.

CONTINUOUS CAST
LAMELLAR STRUCTURE

B.

WARM ROLLED
STRUCTURE

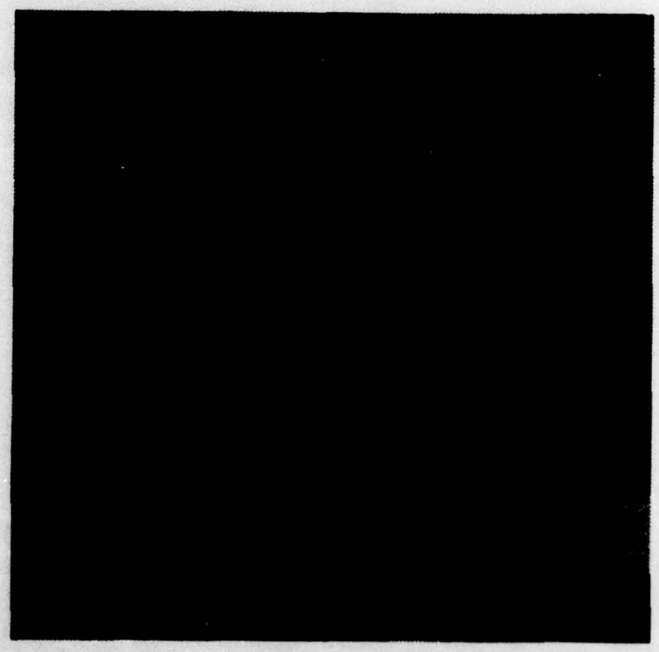


Figure 8 - SCANNING ELECTRON MICROGRAPHS OF AL-17.5Wt.%CU. A. CAST B. WARM ROLLED AT 1200X

1. Specimen Preparation For Microscopy

All specimens were ground, polished, and etched in the same manner, except for fracture surfaces. These specimens were selected and cut from the bulk material with an abrasive, water-cooled, cut-off wheel. They were then ground successively with SiC grit 50 and 320 on grinding belts. The specimens were then hand sanded with emory grit 0 and 3/0, after which they were ready for polishing. Polishing was performed on broadcloth rotary wheels with alumina slurries of 15 micron and .05 micron particles. This procedure resulted in a highly polished specimen surface ready for etching.

Several etchants were available for Al-Cu alloys. Initially Kellers etchant was used (Ref. 3), but it proved difficult to distinguish the two phases. For that reason, 5% HF acid in water was used. This etchant, however, did not show the aluminum grain boundaries. With this etchant, preferably over a week old, 20 to 25 seconds of submergence was sufficient for an effective intermetallic etch. Whenever a fresh batch of etchant was used, extensive over-etching resulted. This effect will be seen in later scanning electron micrographs.

2. Microscopic Observations

Optical microscopy was conducted on Bausch and Lomb and Zeiss optical microscopes in the material science laboratory, at magnifications up to 800X. The Zeiss optical microscope, equipped for polarized light, revealed no additional aspects of the alloy. Optical microscopy was abandoned when the microstructure of the specimens became too fine to be adequately observed at 800X magnification.

Scanning electron microscopy allowed much greater detail of the alloy to be observed. The scanning electron microscope in the material science laboratory was used for all observations and photographs. Initially, the Al_2Cu phase was differentiated from the aluminum phase by energy dispersive x-ray spectroscopy, and it was discovered that the intermetallic appears lighter in scanning electron micrographs. Since all specimens were polished and etched, the largest aperture was used which gave less depth of field but better resolution. Scanning electron microscopy proved to be very helpful in observing the microstructure at up to 8000X magnification and thereby helped in correlating the properties to the microstructure.

D. MECHANICAL TESTING

After the alloy had been cast, warm rolling was accomplished, and microscopy had revealed the desired microstructure, it was possible to investigate the resulting mechanical properties. It was decided that compression testing would result in a greater data base for the limited material and time. Additionally, compression test specimens were manufactured more easily than other specimen configurations. Specimens were machined as 0.25 cm cubes. Compression tests were conducted on an Instron mechanical testing machine, with an attached oven for elevated temperature testing. This equipment is shown in figure 9. Two types of testing were conducted. Room temperature compression tests of the cast specimens and the warm rolled specimens were conducted for mechanical property comparison. Secondly, elevated temperature compression tests were performed only on the warm rolled specimens to investigate some of the more refined areas of the alloy's behavior, such as strain rate sensitivity, activation energy, and superplasticity.

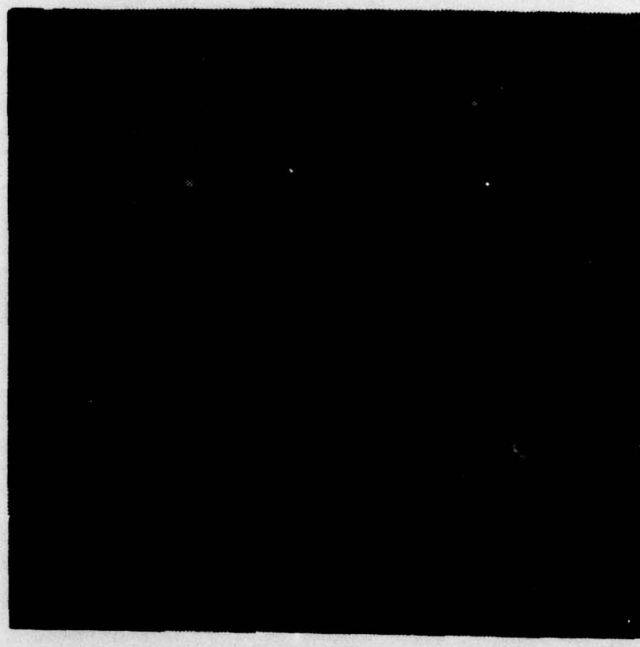


Figure 9 - THE INSTRON MECHANICAL TEST MACHINE IN THE COMPRESSION TEST CONFIGURATION WITH THE OVEN ATTACHED.

1. Room Temperature Compression Testing

Room temperature compression tests were conducted to determine stress-strain behavior of the cast specimens and the warm rolled specimens for comparison. Six specimens of each microstructural condition were tested, three in the longitudinal direction and three in the transverse direction relative to the casting or rolling axis. This procedure indicated that both the as cast and the warm rolled materials were essentially isotropic. In addition to these tests, specimens were tested at six different crosshead speeds at room temperature to investigate the strain rate sensitivity.

2. Elevated Temperature Compression Testing

Elevated temperature compression tests were performed on the warm rolled specimens only. These tests were conducted at six different crosshead speeds: 0.0051, 0.0127, 0.0254, 0.051, 0.127, and 0.254 cm per minute. Additionally, nine temperatures were used ranging from room temperature to 750°K. These tests were conducted to evaluate stress-strain behavior at elevated temperature, strain rate sensitivity as a function of temperature, and activation energy as a function of temperature and flow stress.

3. Special Considerations

During compression testing of any moderately ductile material it is necessary to lubricate the specimens end surfaces. This is done to minimize the barrelling of the unloaded sides which is an indication of a triaxial state of stress. These unwanted stresses were almost completely eliminated in this research by using teflon tape at low and medium temperatures and molybdenum disulfide spray at high temperatures. A triaxial stress state developes as the specimen length dimension becomes small compared to the cross section. This effect was eliminated by ignoring any data beyond an inflection point on the stress-strain curve, characteristic of this phenomenon. Usually, this inflection point occurred at a true strain of approximately 130%. Since the Instron testing machine relies on the deflection of a beam for load measurements, compensation must be made for this deflection in order to obtain accurate measurement of change in specimen length. The test machine was loaded with no specimen involved, and a spring constant of 454,000 Newtons per cm was recorded. The resultant machine deflection was subtracted from recorded specimen deflection. This procedure resulted in more accurate measurements of specimen length and cross section. The modulus of elasticity could not be measured accurately, however, with only this correction.

4. Data Reduction

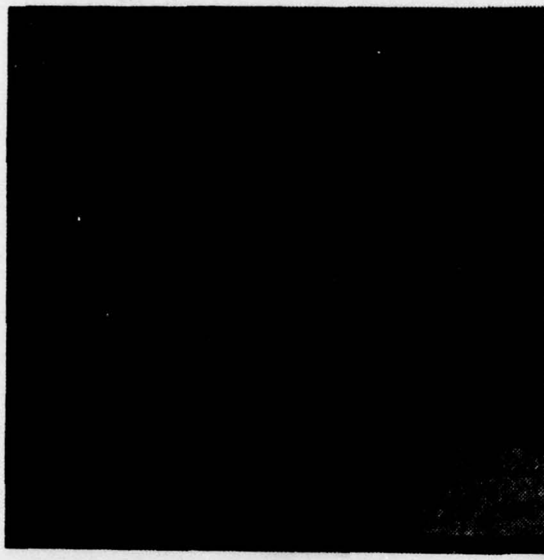
Data was recorded on an automatic chart recorder calibrated for the tests. This record gave load as a function of time, which was converted to true stress versus true strain. Conversion was accomplished by listing load and net change of length for the specimen and dividing load by instantaneous cross sectional area. These results were plotted, and the flow stress at a strain of 15% was used in all subsequent elevated temperature calculations.

III. RESULTS AND DISCUSSION

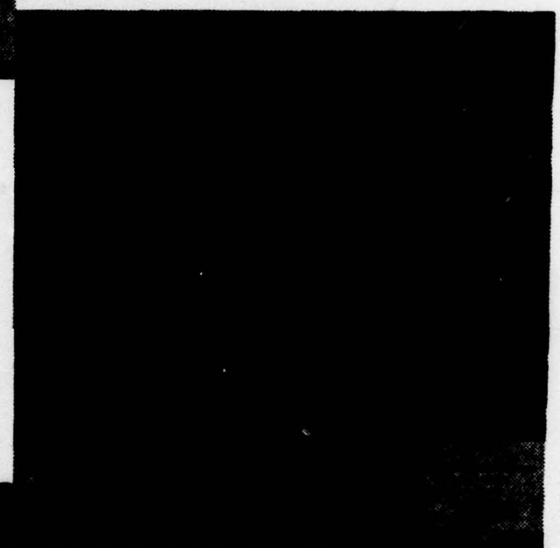
The warm rolled Al-17.5wt.%Cu alloy had a very fine, dispersed microstructure wherein the Al_2Cu intermetallic particles were found to average about 1.2 microns in diameter. This breaking-up of the skeletal structure of the cast Al_2Cu led to a greatly increased strain to fracture but reduced the yield stress. The maximum compressive stress was only slightly effected, and hence there was a six-fold increase in toughness, as inferred from the area under the stress-strain curve. Neither the cast samples nor the warm worked samples exhibited any significant strain rate dependence of the flow stress at room temperature; also no anisotropic behavior was observed. The warm worked material did display strain hardening whereas the cast material did not. The cast specimens failed due to the cracking of the intermetallic skeletal structure, while the warm worked material had no such structure wherein cracking would occur. At elevated temperature the flow stress of the warm rolled material was found to be dependent on both temperature and strain rate. At high temperatures the strain rate sensitivity was found to approach values indicative of superplasticity. The calculated activation energies show three distinct regions of behavior, two of which correspond to mechanisms associated with pure aluminum. The third region, at high temperature and low stress, was also indicative of the onset of superplastic behavior. This combination of high room temperature toughness and superplasticity was considered a significant improvement.

A. MICROSTRUCTURAL CHARACTERIZATION

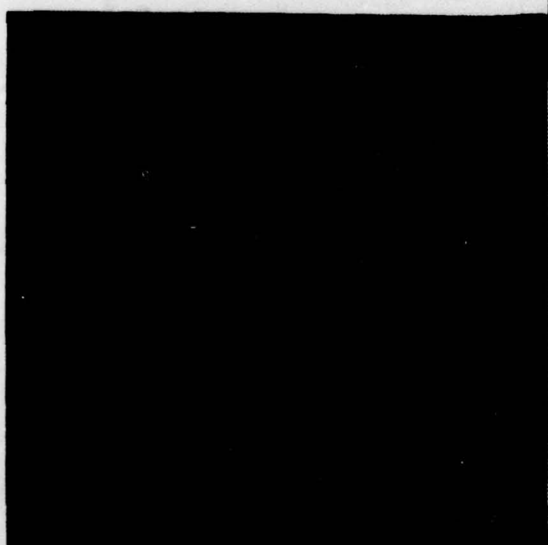
The effectiveness of warm working to refine the microstructure is dependent on temperature, strain rate, and strain (Ref. 1). The finest particle size would be achieved when the highest strain rate possible was used at the lowest temperature permissible to induce as much strain as possible. Figure 10 shows the microstructure of the warm rolled Al-17.5wt.%Cu alloy at magnifications of 1300X, 2600X, and 8000X. The average size of the Al_2Cu intermetallic particles was calculated to be 1.2 microns by using the average lineal intercept method. Although this is a relatively fine particle size, it is apparent that an even finer microstructure could be achieved if it were broken into more of the sub-micron particles revealed at 2600X and 8000X magnification. It is generally accepted that this refinement takes place by shearing of the intermetallic grain boundaries and plastic flow of the grains in general. These mechanisms are controlled during warm rolling by the temperature and the strain rate. Additionally, grain growth is controlled by temperature. It is apparent that a lower temperature would result in a finer grain size for a given strain rate. The grain refinement shown in figure 10 is by no means the ultimate. A greater degree of control over temperature, strain rate, and strain would be necessary to achieve that goal.



A.
1350X



B.
2600X



C.
8000X

Figure 10 - SCANNING ELECTRON MICROGRAPHS OF THE WARM ROLLED AL-17.5Wt.%CU ALLOY AT VARIOUS MAGNIFICATIONS.

B. ROOM TEMPERATURE PROPERTIES

Figure 11 is a plot of true stress versus true strain for the as cast material and the warm rolled material for tests at room temperature. These are representative curves for each material. True strain was terminated at 100%, although some warm worked specimens displayed strains in excess of 140% before triaxiality voided the results. The hardness of each material was measured on the Rockwell B scale. As-cast specimens exhibited hardness in the range of Rb 55 to 60, with the warm worked specimens showing approximately half that value. The yield point and maximum compressive stress for the cast material were 321 MPa and 419 MPa. The yield point for the warm rolled material was 236 MPa. In both cases the modulus of elasticity, although similar, was in error due to inaccuracies in computing machine stiffness and settling. However, this had little influence on other calculations. The maximum compressive stress was taken at a strain of 100%. Beyond this strain, triaxial conditions could influence the results. Additionally, it must be remembered that these results were based on compression tests, where necking had no influence. The maximum compressive stress for the warm rolled material was 368 MPa at a strain of 100%. The strain-to-fracture was one of the most important measures of success in warm rolling. The cast specimens averaged a strain-to-fracture of 16% whereas the warm rolled specimens rarely failed at all. Toughness of a material is proportional to the area under the stress-strain curve. Using this measure of toughness and a final strain of 100% for the warm rolled material, there was a six-fold increase in toughness produced by the warm working.

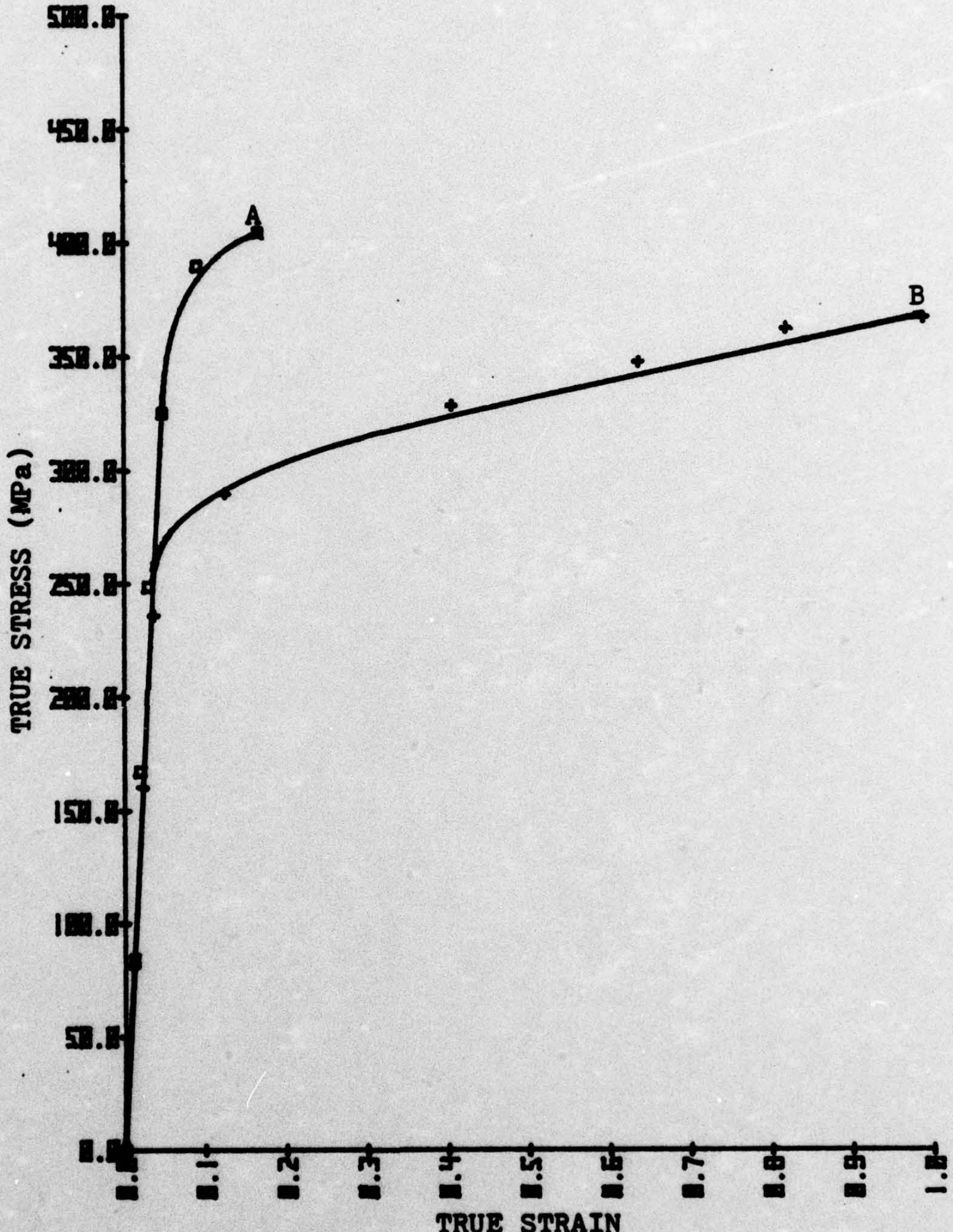


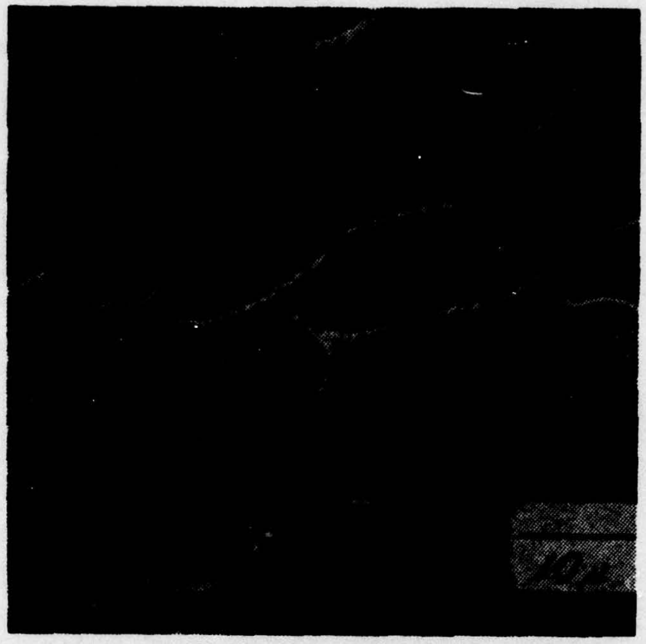
Figure 11 - TRUE STRESS - TRUE STRAIN CURVES FOR: A. AS CAST SPECIMENS B. WARM ROLLED TO 150% STRAIN.

Neither the as cast specimens nor the warm worked specimens exhibited any degree of anisotropy in compression testing. Figures 12 and 13 are scanning electron micrographs of the microstructures of the longitudinal and transverse directions of each material. These micrographs correlate with the test results of isotropic behavior.

The warm worked specimens exhibited strain hardening which was absent in the as-cast condition. This strain hardening was assumed to obey a simple power law curve.

$$\sigma = K\varepsilon^{N} \qquad (2)$$

N is the strain hardening exponent, and K is a material constant. When plotting the logarithm of stress versus the logarithm of strain, the strain hardening exponent, N, is the resultant slope. Figure 14 is such a plot which shows data points for all warm rolled specimens at room temperature for strains from 4% to 100%. A least squares linear regression was performed, and the slope was found to be 0.134. This would indicate that the onset of necking in tension would occur at a strain of 13.4% and failure at some higher strain (Ref. 4). This value is within the range of commonly used structural materials. The brittle cast material had been refined to exhibit strain hardening and more ductility.



A.
LONGITUDINAL AT 1600X

B.
TRANSVERSE AT 1200X

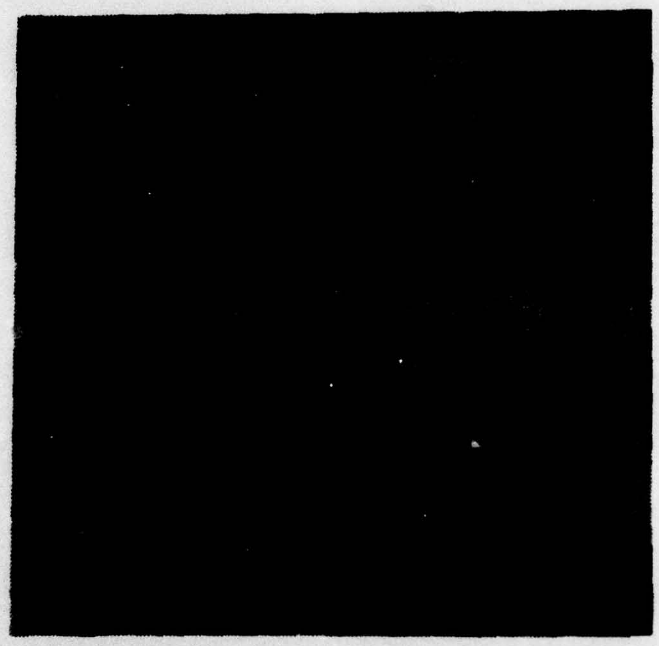
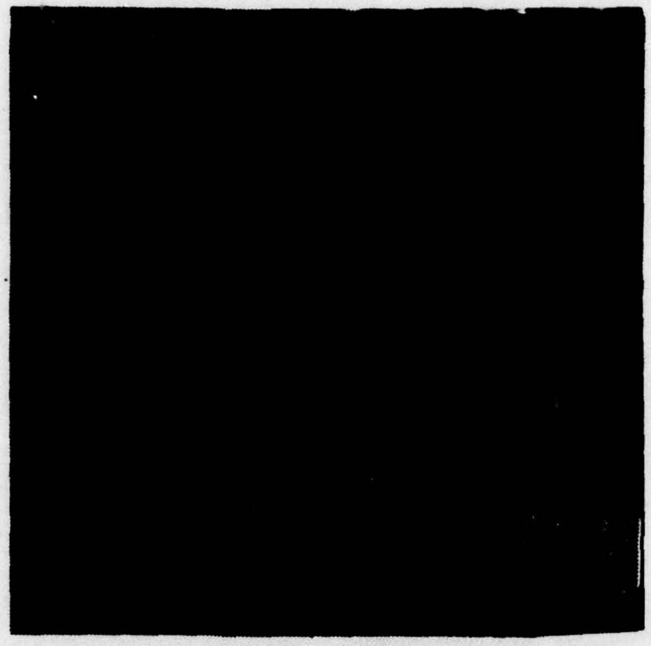


Figure 12 - SCANNING ELECTRON MICROGRAPHS OF AL-17.5Wt.%CU ALLOY IN THE AS CAST CONDITION.



A.
LONGITUDINAL AT 1300X

B.
TRANSVERSE AT 1200X

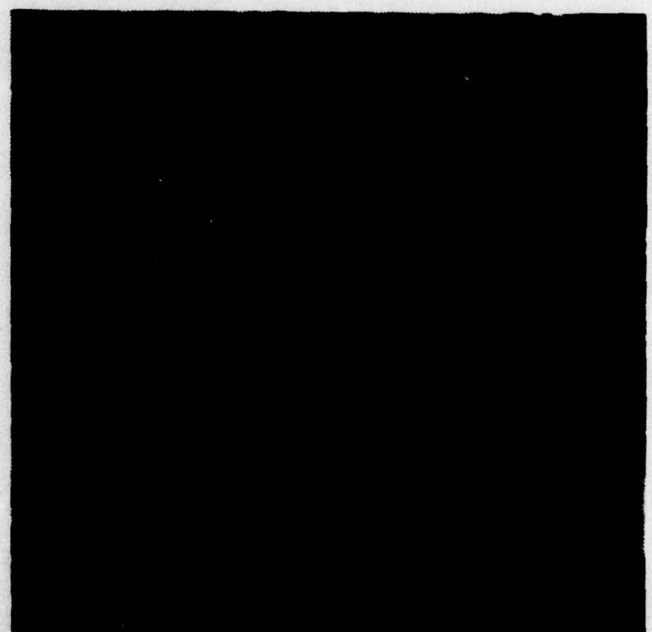


Figure 13 - SCANNING ELECTRON MICROGRAPHS OF AL-17.5Wt.%CU ALLOY AFTER WARM ROLLING TO A TRUE STRAIN OF 150%.

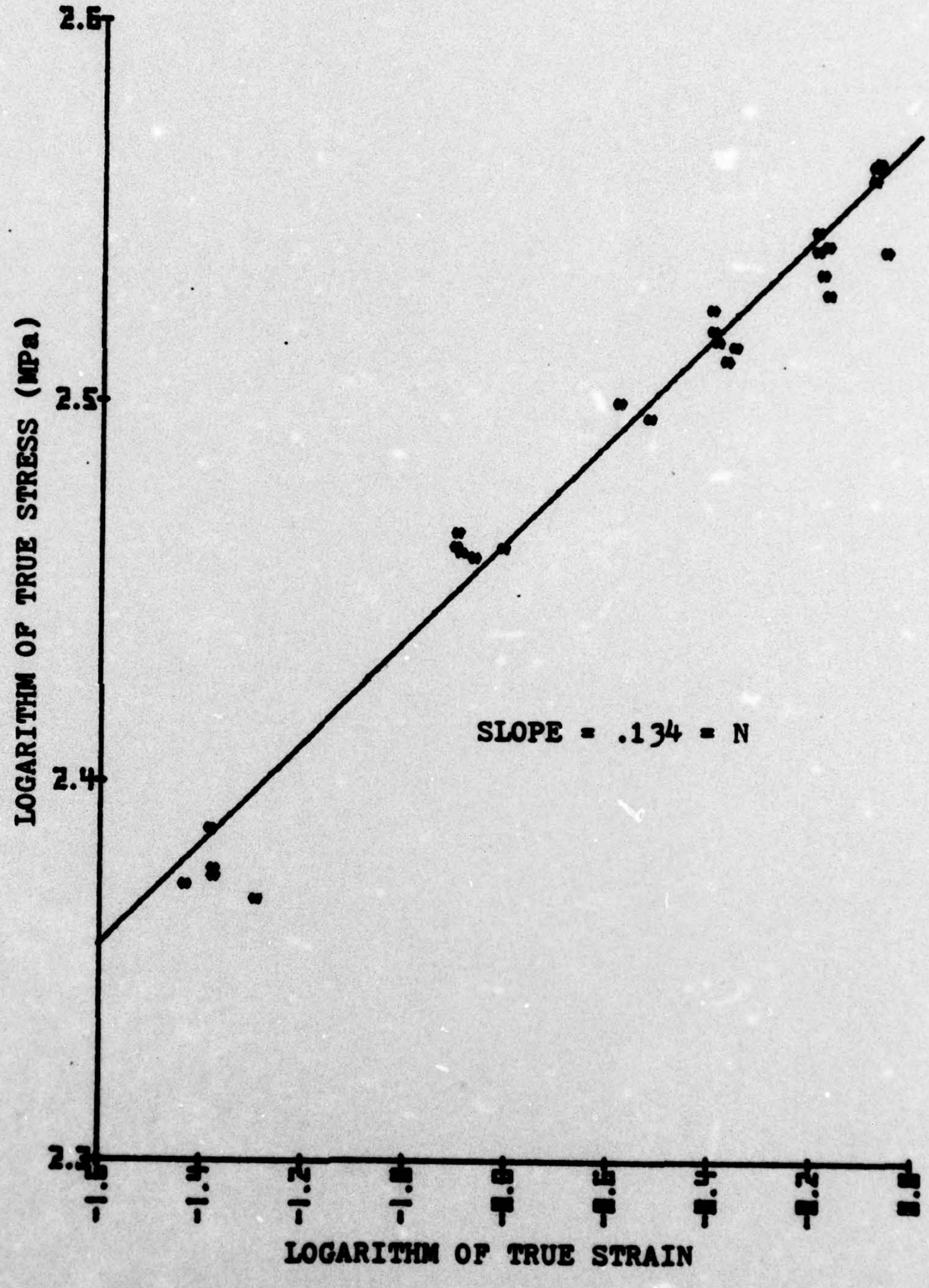
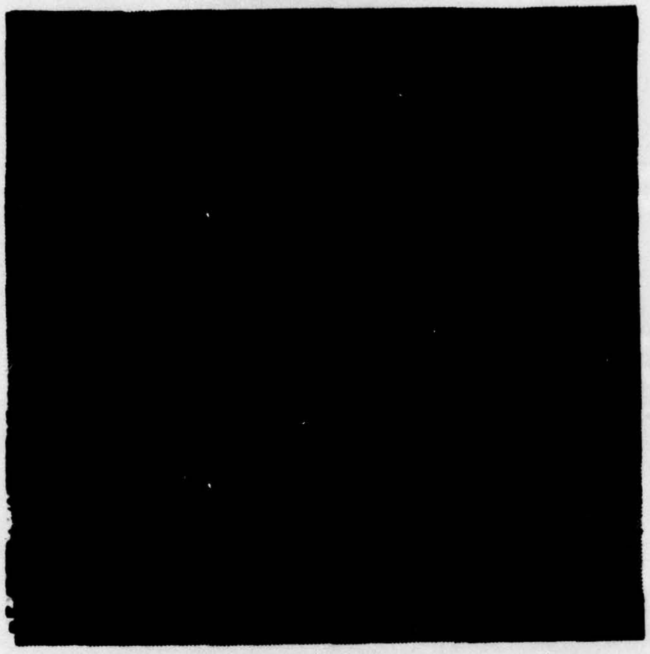


Figure 14 - LOGARITHM OF STRESS - LOGARITHM OF STRAIN PLOT FOR THE WARM ROLLED CONDITION.

One final note about the room temperature properties is in order. The failure mechanism of the as-cast specimens was revealed by the scanning electron microscope. Figure 15A shows the microstructure of an as-cast specimen that failed during a compression test at room temperature. The mechanism of failure was evidently crack propagation through the brittle intermetallic skeletal structure. Many cracks can be seen in this micrograph. Figure 15B is presented for comparison. This is a scanning electron micrograph of a warm worked specimen that was tested in compression and did not fail. It lacks the brittle structure that caused the as-cast material to fail.

Generally, the warm worked material possessed properties at room temperature that were more amenable to engineering applications than the as cast material. Enhanced toughness was the major asset. It was obtained at the expense of yield strength and hardness. The warm worked material also exhibited strain hardening which was an added benefit. The final product of this research possessed moderate strength and high ductility at room temperature.



A.
AS CAST AT 1600X

B.
WARM ROLLED AT 1400X

Figure 15 - SCANNING ELECTRON MICROGRAPHS OF AL-17.5Wt.%CU ALLOY AFTER COMPRESSION.

C. ELEVATED TEMPERATURE PROPERTIES

It was assumed at the outset that flow stress was a function of strain rate and temperature. This was not unwarranted since aluminum exhibits such behavior. It was assumed, in particular, that this behavior would follow the equation given (Ref. 5).

$$\dot{\varepsilon} = K(\sigma/E)^{1/M}\ \mathrm{EXP}(Q/RT) \qquad (3)$$

Sigma over E is the modulus-compensated stress, M is the strain rate sensitivity exponent, Q is the activation energy, T is the absolute temperature, R is the gas constant, and K is a material constant. The strain rate sensitivity exponent, M, was found by holding temperature constant. It varied from 0.0 at room temperature to 0.27 at 755°K. This latter value is indicative of the onset of superplasticity. M values of 0.3 or greater are associated with superplastic behavior (Refs. 1 and 6). The activation energy, Q, was found by holding modulus-compensated stress constant. Values obtained for activation energy also indicated the onset of superplastic behavior.

1. **Flow Stress as a Function of Temperature**

The compressive stress-strain curves obtained at temperatures from 300°K to 755°K were analysed to generate the flow stress-temperature curves shown in figure 16. The flow stress plotted was the stress at a true compressive strain of 15%. Each of the six strain rates, corresponding to the six crosshead speeds, is represented by a single curve. As suggested by equation 3, the flow stress was inversely proportional to temperature and directly related to strain rate.

2. **Strain Rate Sensitivity Exponent**

Assuming again that equation 3 would describe this material behavior and holding temperature constant, the strain rate sensitivity exponent, M, would be the slope of the logarithms of stress versus the logarithms of strain rate plot. Figure 17 shows this data plotted for each temperature. A least squares linear regression was performed for each temperature which resulted in the strain rate sensitivity exponents as a function of temperature. These values are shown in figure 18. The exponents approach a value of 0.11 at 550°K and then approach a value of 0.3 as the temperature approaches the melting point. This latter value is indicative of the onset of superplastic behavior.

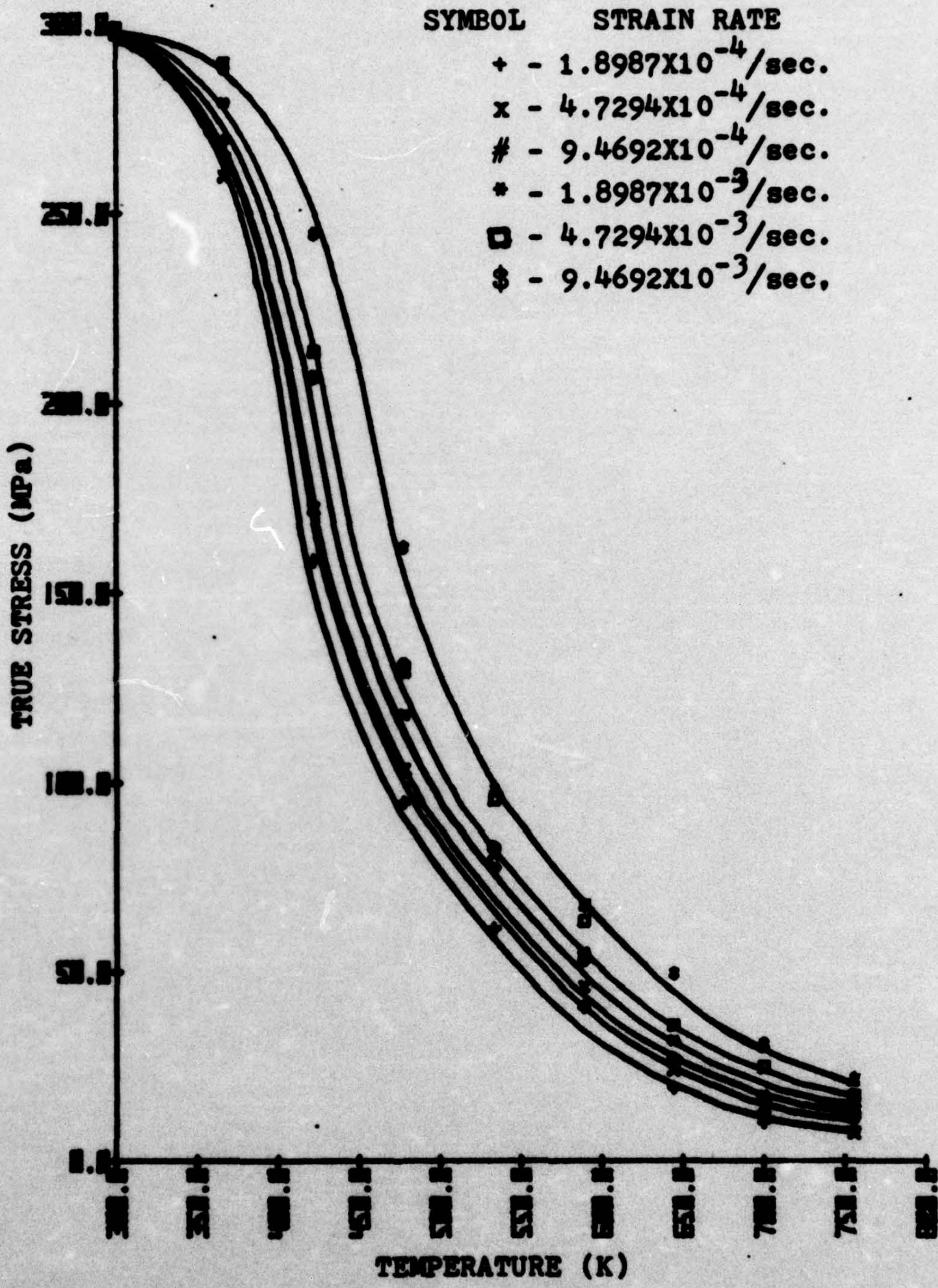


Figure 16 - FLOW STRESS - TEMPERATURE PLOT FOR A TRUE STRAIN OF .15 FOR ALL SIX STRAIN RATES.

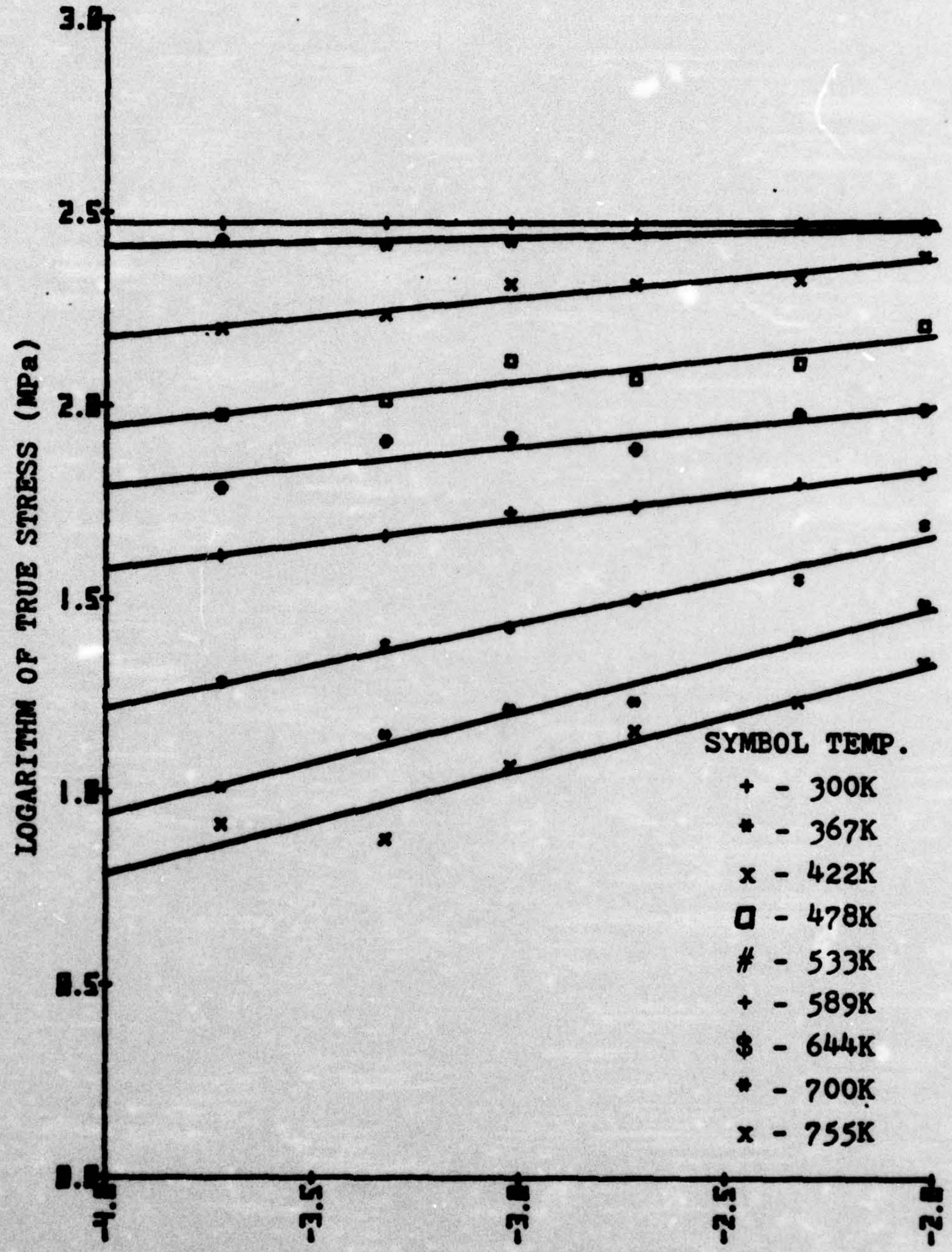


Figure 17 - LOGARITHM OF FLOW STRESS VERSUS THE LOGARITHM OF STRAIN RATE AT A CONSTANT STRAIN AND TEMPERATURE.

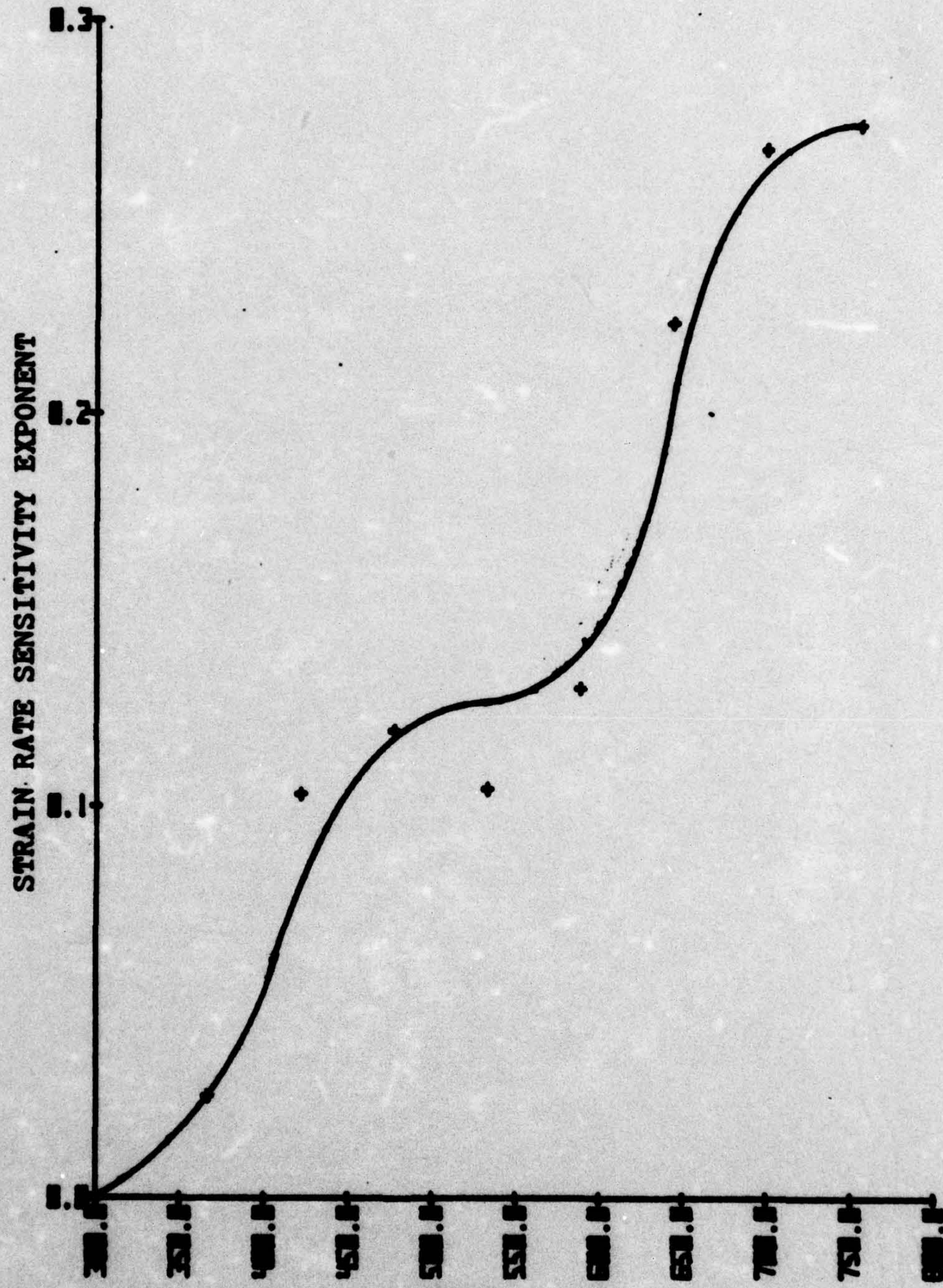


Figure 18 - STRAIN RATE SENSITIVITY EXPONENT, M, AS A FUNCTION OF TEMPERATURE.

3. <u>Activation Energy</u>

Figure 19 shows the modulus of elasticity, E, of aluminum as a function of temperature (Ref. 7). In order to eliminate the temperature dependence of the elastic modulus from consideration and thereby obtain a true activation energy for deformation, the modulus-compensated flow stress was plotted as a function of temperature as shown in figure 20. This figure is very similar to figure 16 but shows a greater variation of compensated flow stress between varying strain rates.

Figure 20 was then used to extract temperature - strain rate data for constant modulus-compensated stress. The natural logarithm of the strain rate was plotted as a function of reciprocal temperature in figure 21 from this data. From equation 3, it was observed that the resultant straight line slope would be equal to the activation energy divided by the gas constant. Twenty-two different values of modulus-compensated stress were used ranging from 0.00035 to 0.004. Each of these values resulted in one activation energy; only some resultant curves are shown in figure 21. In all cases a least squares linear regression was performed to obtain the best slope. A distinct change of slope is observed in figure 21.

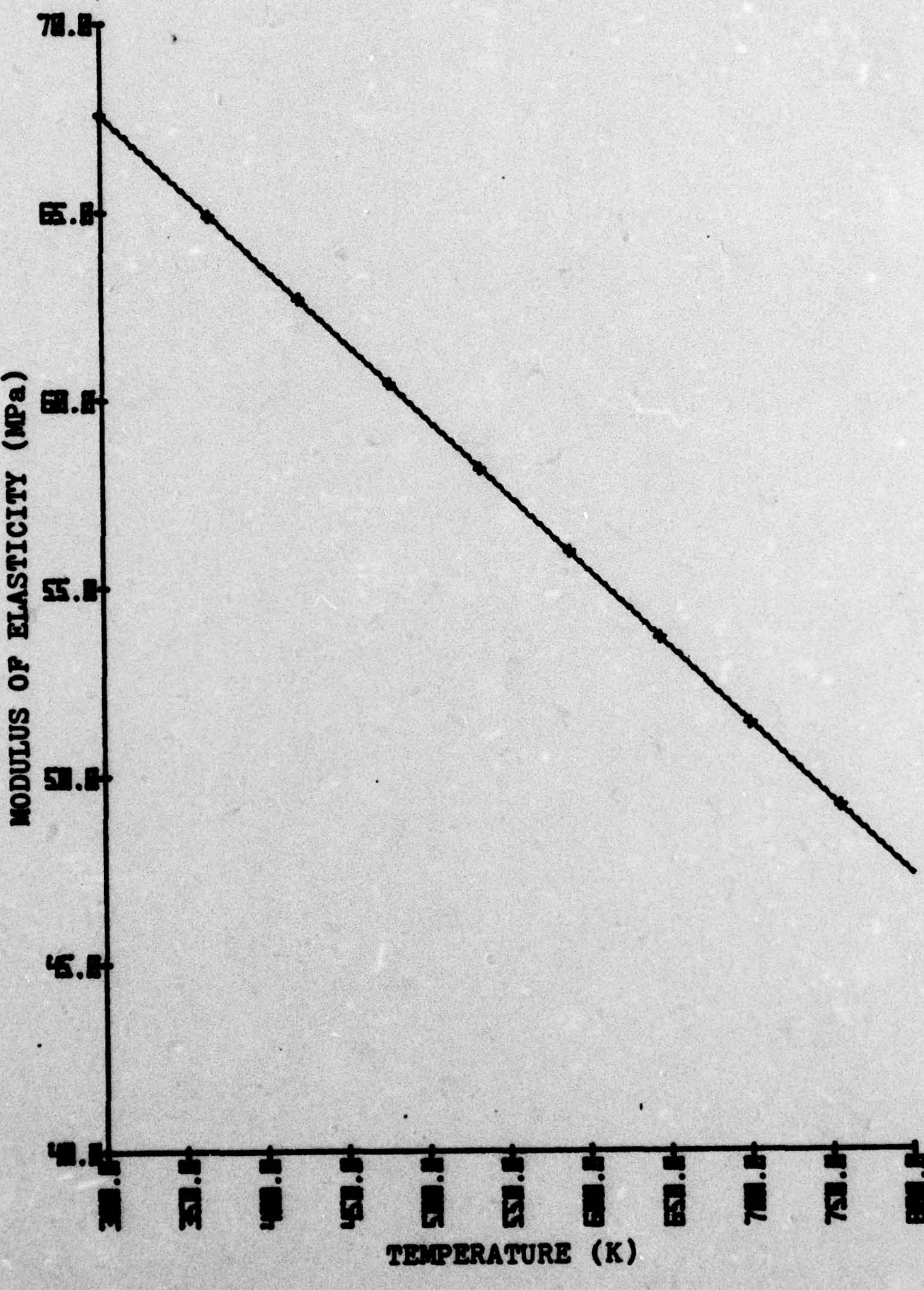


Figure 19 - MODULUS OF ELASTICITY VERSUS TEMPERATURE FOR ALUMINUM FROM REFERENCE (7).

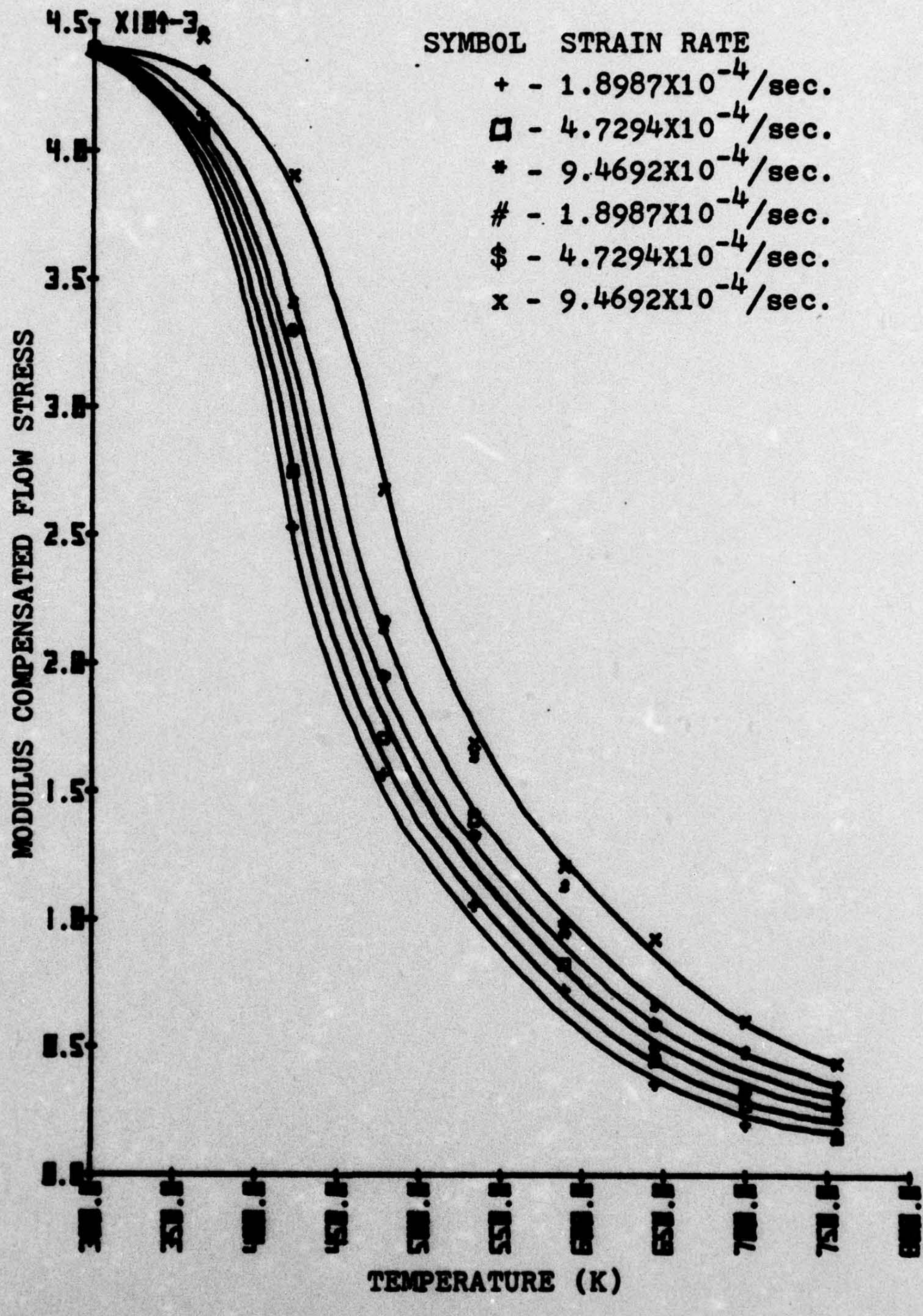


Figure 20 - MODULUS-COMPENSATED FLOW STRESS VERSUS TEMPERATURE FOR THE SIX DIFFERENT STRAIN RATES APPLIED.

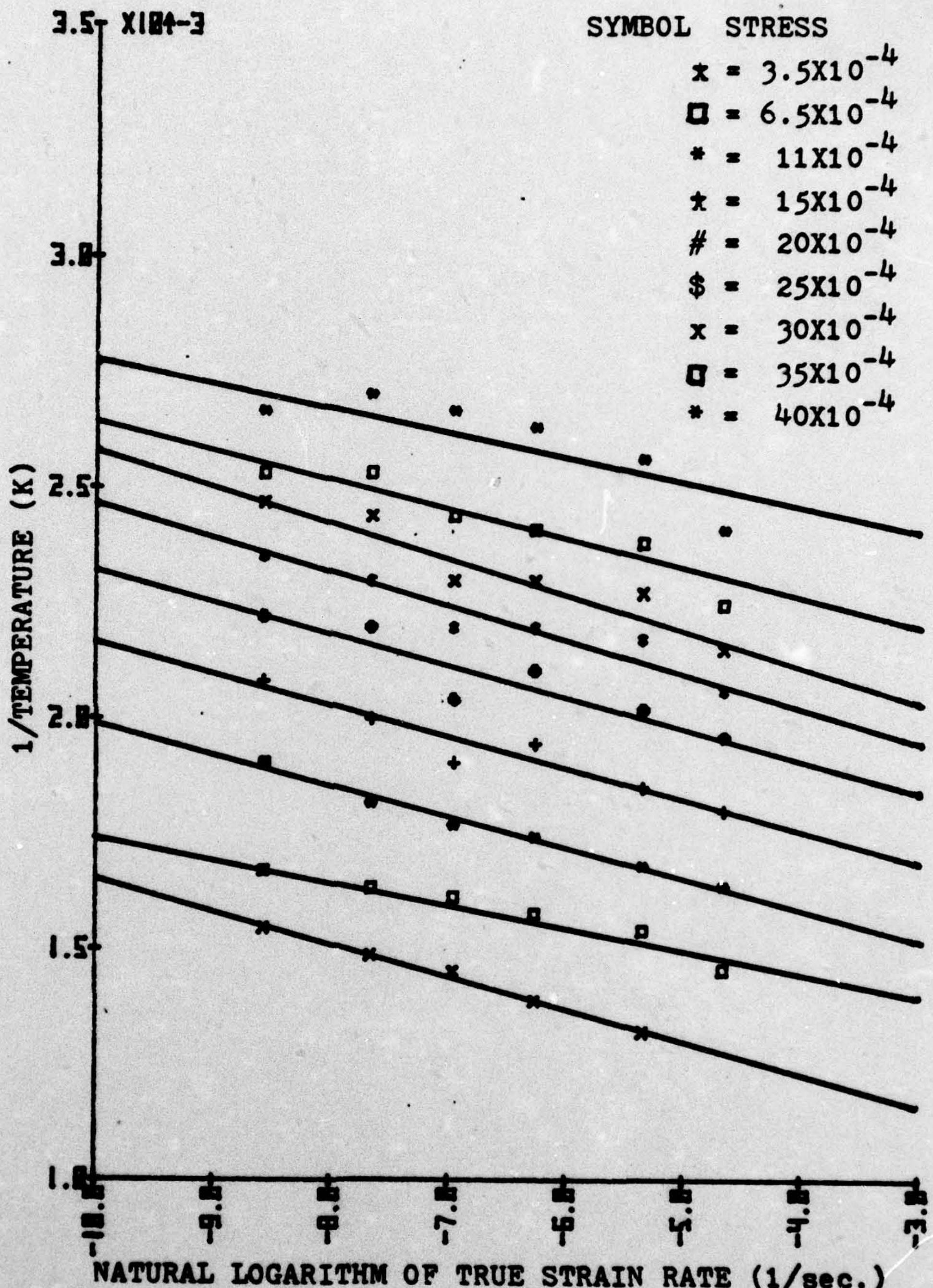


Figure 21 - NATURAL LOGARITHM OF THE STRAIN RATE VERSUS THE INVERSE TEMPERATURE FOR A CONSTANT COMPENSATED STRESS

The activation energies obtained from figure 21 were plotted as a function of modulus-compensated stress, as shown in figure 22. This curve shows three distinct regions of behavior. At high modulus-compensated stress, above 0.0012, the activation energy was nearly constant with a value of 27 KCal per mole. This was roughly the value that is associated with pipe diffusion in pure aluminum (Ref. 5). At compensated stresses between 0.0006 and 0.001 the activation energy rose to a value of 37 KCal per mole. This value correlates well with the activation energy for self diffusion in pure aluminum (Ref. 8). In pure aluminum these are the only two regions found. The drop observed in figure 22 at modulus-compensated stresses below 0.0006 has no correlation with mechanisms in pure aluminum. This decrease in activation energy is , however, associated with the onset of superplasticity (Ref. 4).

The activation energy was plotted as a function of temperature for three different strain rates in figure 23. Here again three regions of behavior were observed. Additionally, a greater strain rate causes the temperature to be higher before the mechanisms change. At the lowest strain rate, the transition (to the activation energy associated with the onset of superplasticity) occurs at a much lower temperature than at the higher strain rates.

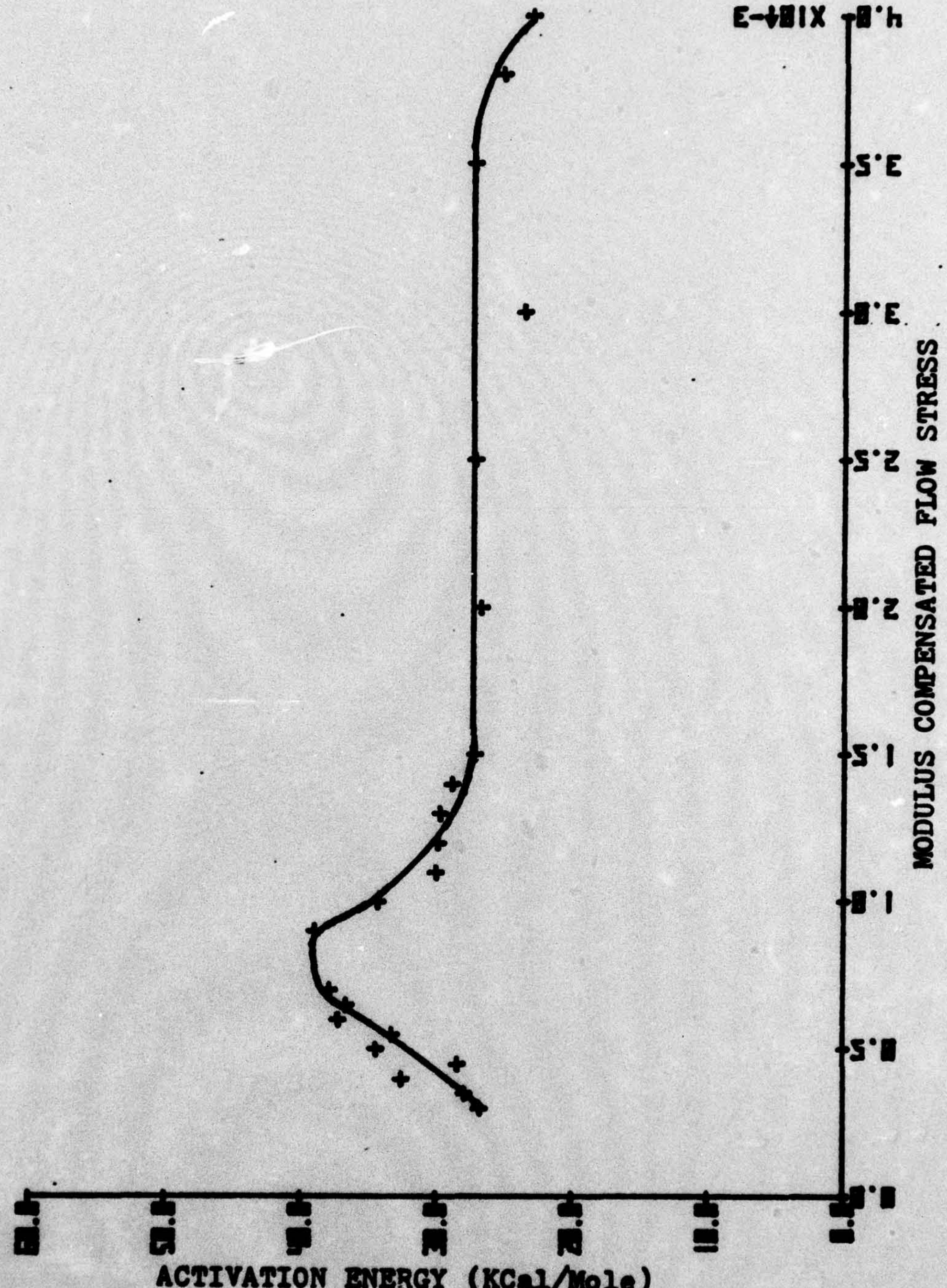


Figure 22 - THE ACTIVATION ENERGY AS A FUNCTION OF THE MODULUS-COMPENSATED FLOW STRESS.

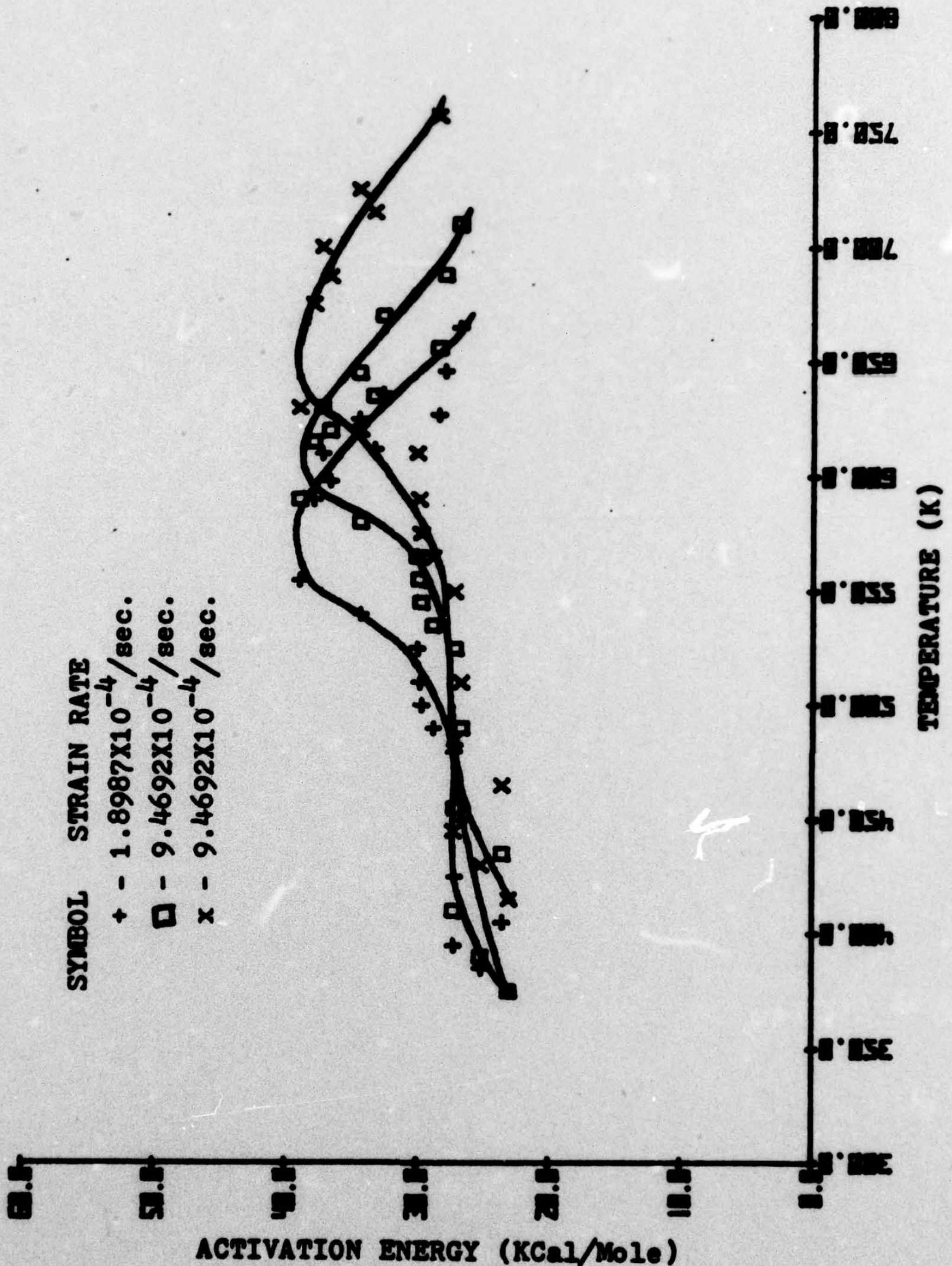


Figure 23 - THE ACTIVATION ENERGY AS A FUNCTION OF TEMPERATURE FOR THREE DIFFERENT STRAIN RATES.

D. DISCUSSION

A nearly ideal engineering material would have high room temperature strength, with strain hardening and high toughness, but it would be easily formable at elevated temperatures. This latter property has been the object of extensive research into superplasticity. Few material scientists have investigated both room temperature properties and superplasticity. This discussion will present current research in these areas and the expectations of further research.

Room temperature properties should be a major concern in any research effort. This has not been the case, however, in efforts to achieve and understand superplasticity. Yield stress is increased by several methods. Three of these methods apply to microstructural refinement of a eutectic binary alloy. The Hall-Petch relationship shows that yield strength is inversely proportional to the grain size (Ref. 9).

$$\sigma_y = \sigma_i + Kd^{-1/2} \qquad (4)$$

Sigma Y is the yield stress, Sigma i is the average yield strength of a single grain, and K is a material constant. The grain size is given by d. This indicates that a finer grain size, which could be achieved by more warm rolling, would result in a greater room temperature yield strength. The Zener-McLean relationship indicates that a smaller particle size and a greater intermetallic volume fraction would decrease the overall grain size (Ref. 10).

$$d = 4R/3F \qquad (5)$$

Here, F is the volume fraction of intermetallic and R is the average intermetallic particle size. The grain size , d, is the result. This relationship indicates that the grain size can be reduced by increasing the volume fraction or reducing the particle size and results in a greater yield strength according to the Hall-Petch relationship. A third way to improve the room temperature properties is shown by assuming a dispersion strengthening relationship (Ref. 9).

$$\sigma_y = \sigma_0 + 4Gb/l \qquad (6)$$

The closest distance between particles is l, The closest distance between particles is l, b is the burgers vector, and G is the shear modulus. This relationship indicates that the yield stress is inversely proportional to the intermetallic particle spacing. The specific relationship for the warm rolled microstructure is unknown; however, all such theories predict higher strength for a finer microstructure. Summarizing, the room temperature yield stress would be expected to increase with finer grain size, greater intermetallic volume fraction, and a greater number of intermetallic particles.

Presently, only one major research effort has been made to correlate room temperature properties to superplasticity. This research was done by Sherby on ultra-high-carbon steels (Ref. 1). This work produced what might be classed as a super metal. At room temperature it has the strength and ductility of expensive super alloys, but at high temperature it is superplastic. No complete research of Al-Cu has been done. Cahccn (Ref. 6) did report a room temperature yield strength of 220 MPa in tension which compares well with these results of 236 MPa in compression. He also reported an ultimate tensile strength of 341 MPa at 19% elongation. Since this research was done in compression, this compares well with results of 315 MPa at a true strain of 0.2. There is a need for more complete research in the area of room temperature properties as a function of grain size and composition in the warm worked state.

Superplasticity has been a subject of major research for several years. The ability of a material to form easily into complex shapes has become increasingly important. One of the most widely accepted requirements for this to occur is ultrafine grain size (Refs. 1, 6, and 11). Additionally, spherical grains seem to further enhance superplasticity (Ref. 1). Although this phenomenon is being investigated intensely, few researchers agree on the exact mechanism for this behavior. Ashby and Verral (Ref. 12) have modeled data for superplastic behavior, claiming grain boundary sliding with diffusional accommodation as the mechanism. Most agree that grain boundary sliding or shearing is involved but claim other accompanying mechanisms (Ref. 11 and 13). The overriding importance of grain size is acknowledged in all theories.

Other researchers of the Al-Cu system have reported findings consistent with the results obtained in this research. Cahoon (Ref. 6) produced a slightly finer microstructure which resulted in strain rate sensitivity exponents that approached 0.4. This compares well with the values approaching 0.3 achieved in this research program. Holt and Backhofen (Ref. 11) worked with a 33wt.%Cu alloy which resulted in strain rate sensitivities approaching 0.65. This value indicates complete superplastic behavior. Only Cahoon reported some limited results for room temperature properties.

It would seem that both room temperature properties and superplasticity can be enhanced by the same means. Increasing the volume fraction of intermetallic and decreasing the grain size will achieve this end. Warm microstructure as possible would result in high yield strength at room temperature and extensive superplasticity at high temperature.

IV. CONCLUSIONS AND RECOMMENDATIONS

The facilities at the material science laboratory were sufficient for refining the aluminum-copper microstructure through warm rolling. Although close control could not be exercised over strain rate or temperature, there was sufficient range in the alloy characteristics to overcome this shortcoming. The microstructure that resulted was composed of a fine dispersion of intermetallic particles in an aluminum matrix.

The room temperature properties which resulted from this refined microstructure were an improvement over the as-cast properties. This improvement was primarily in enhanced ductility and the resultant increase in toughness. No other researchers have reported these results, or for that matter, have investigated these areas.

The onset of superplasticity was obtained in the warm rolled material. This has been the major effort of other researchers. The results obtained in this research correlate well with these other research efforts.

Although an analysis of room temperature properties versus high temperature properties for various compositions and warm worked conditions was not accomplished, the following effects would be anticipated. Yield strength would increase with a finer microstructure. Ductility and toughness would continue to increase with a greater degree of warm working. Superplasticity at elevated temperature would continue to increase as more copper is added and a finer grain size is achieved.

It is recommended that a heated extrusion press be used in future warm working to insure the the greatest control possible over the warm working conditions. In this way the required microstructure could be achieved and the resultant properties analysed. This method would also produce billets readily usable for tensile testing, thus avoiding some of the pitfalls of compression testing. The original goal of this research program was not achieved. The ground work for further research, however, has been laid. It is recommended that this research continue with the hope that an ultimate warm working procedure can be developed which will result in high room temperature strength, ductility, and toughness and superplasticity at elevated temperatures.

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