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A Final Report
Grant No. N00014-85-K0179

March 1, 1985 - August 31, 1989

INVESTIGATION OF THERMO-MECHANICAL EFFECTS IN
SILICON CARBIDE WHISKER/AL ALLOY COMPOSITES

Submitted to:

Office of Naval Research
800 N. Quincy Street
Arlington, VA 22217-5000

Attention:

Dr. Steven Fishman
Non-Metallic Materials
Code 1131N

Submitted by:

F. E. Wawner, Jr.
Research Professor

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Graduate Research Assistant

Report No. UVA/525398/MS90/104
June 1990

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| REPORT DOCUMENTATION PAGE | | | Form Approved OMB No. 0704-0188 | |
|---|---|--|--|---|
| <small>Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.</small> | | | | |
| 1. AGENCY USE ONLY (Leave blank) | | 2. REPORT DATE June 1990 | | 3. REPORT TYPE AND DATES COVERED Final March 1, 1985 - August 31, 1989 |
| 4. TITLE AND SUBTITLE Investigation of Thermo-Mechanical Effects in Silicon Carbide Whisker/Al Alloy Composites | | | 5. FUNDING NUMBERS N00014-85-K0179 | |
| 6. AUTHOR(S) F. E. Wawner, Jr. R. D. Schueller | | | | |
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) University of Virginia School of Engineering and Applied Science Department of Materials Science Thornton Hall Charlottesville, VA 22903-2442 | | | 8. PERFORMING ORGANIZATION REPORT NUMBER UVA/525398/MS90/104 | |
| 9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Mr. Michael McCracken, Administrative Contracting Officer Office of Naval Research Resident Representative 2135 Wisconsin Avenue, N. W., Suite 102 Washington, DC 20007 | | | 10. SPONSORING/MONITORING AGENCY REPORT NUMBER | |
| 11. SUPPLEMENTARY NOTES | | | | |
| 12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited | | | 12b. DISTRIBUTION CODE | |
| 13. ABSTRACT (Maximum 200 words) <p>This program deals with determining factors that influence strength, ductility, and elongation to failure in SiC whisker/Al alloy composites. Specifically, a microstructural study was made in an attempt to define extrinsic and intrinsic factors that control fracture and thus influence properties.</p> <p>One of the more important products produced by APMC from SiC_w/Al composites has been the rolled sheet. The response of the material to secondary processing and thermo-mechanical treatment is of utmost importance with respect to material quality. The present study emphasizes microstructural-mechanical property characterization of the rolled material.)</p> | | | | |
| 14. SUBJECT TERMS SiC whisker composites; thermomechanical treatment; processing- microstructure-mechanical property correlations (JES) | | | 15. NUMBER OF PAGES 6 + 70 p. appendix | |
| | | | 16. PRICE CODE | |
| 17. SECURITY CLASSIFICATION OF REPORT unclassified | 18. SECURITY CLASSIFICATION OF THIS PAGE unclassified | 19. SECURITY CLASSIFICATION OF ABSTRACT unclassified | 20. LIMITATION OF ABSTRACT unlimited | |

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INTRODUCTION

The stigma of high cost which has inhibited the use of metal matrix composite materials for many years is slowly being removed. Improved manufacturing techniques for the reinforcements and composites, higher volume usage, and a realization that lifetime factors and efficiency improvements must be considered in an overall economic evaluation have led to increased incorporation of the materials into present day and future design concepts.

Additionally, many of the conventional metals and their alloying components (i.e., additives such as Ti, Cr, Co) are primarily imported from potentially unstable nations and have been placed on a critical materials list. For this reason, replacement of these materials may be a forced necessity in the very near future. Metals matrix composites are strongly being considered to fulfill this role.

One such materials system that has moved rapidly from its laboratory inception to commercialization is a silicon carbide whisker reinforced aluminum composite material. This material is composed of typically 15-20 volume percent whiskers in various aluminum alloy matrices and has demonstrated strength values that equal or exceed most Al alloys and a Young's modulus that is 75% higher (18×10^6 psi [124 GPa]). These properties are quite similar to those obtained from many titanium alloys and at a lighter weight, hence, specific properties for the whisker reinforced material are even more impressive. These properties along with

additional advantages of being able to use conventional forming methods such as extrusion, forging, rolling, etc. make the material attractive from an economic as well as a convenience standpoint.

The development of SiC whisker reinforced composite material has followed a logical progression in that baseline mechanical property data has been established, as well as limited microstructural characterization. As with many materials, success comes quickly in the early stages of development only to succumb to more subtle problems later on. These subtleties are overcome only by completely understanding the system. Most frequently the limitations are microstructurally related and on an atomic level. Hence, correlation of specimen history and properties with microstructural observations can lead to an understanding which could extend the range of properties for the material.

This program deals with determining factors that influence strength, ductility, fracture toughness, and elongation to failure in SiC whisker/Al alloy composites. Specifically, a microstructural study is being made in an attempt to define extrinsic and intrinsic factors that control fracture and thus influence properties. The information derived will then be used to make recommendations for upgrading the properties of this important materials system.

Past microstructural studies have indicated that constituent particles in the 3-5 micron size range dominate failure in the present composite systems of interest (i.e. SiC/2124 and

SiC/6061). These particles, identified through X-ray analysis as Al_2CuMg , $\text{Al}_{20}\text{Mn}_3\text{Cu}_2$, and FeCuMnAl_6 , definitely influence fracture toughness since they generally are brittle and form in critical locations, such as along whisker-matrix interfaces and along grain boundaries. These particles are also found to form in areas where whiskers are in contact.

Minimizing these detrimental particles is a first step toward improved fracture toughness. Improved extrusion and rolling methods developed at Advanced Composite Materials Corporation have greatly improved whisker distribution throughout the matrix. With better whisker distribution, the probability of precipitate formation at whisker-whisker contact areas is greatly decreased. In addition to minimizing precipitate formation sites, matrix alloy chemistry has been altered to reduce the amount of elements present (i.e. Fe, Mn, Mg, Cu), which are the major components of these constituent particles.

One of the more important products produced by ACMC from SiC_w/Al composites has been the rolled sheet. The response of the material to secondary processing and thermo-mechanical treatment is of utmost importance with respect to material quality. The present study emphasizes microstructural-mechanical property characterization of the rolled material.

PROGRAM SUMMARY

It has been confirmed in this study that when Al₂124/15% SiCw is hot rolled it loses a good portion of its strength. It was found that this loss in strength is mainly due to overaging of the precipitation hardenable matrix. Evidence shows that large Al₂Cu precipitates grow as the material is allowed to cool down after the rolling step.

It was also confirmed that the strength of the material can be regained by subjecting it to a T6 heat treatment (1 hour at 500°C, CWC, and aged at 150° for 8 hours). Experiments show that the strength is regained mainly because the heat treatment disperses the precipitates. It was determined that the normal T-6 heat treatment times can be reduced due to enhanced diffusivity resulting from small grain size, (hence a large number of grain boundaries) and the high dislocation density of this material. The precipitates can be dispersed by solutionizing the material at 500°C for very short periods of time (as short as 1.5 minutes) followed by cold water quenching. Likewise, it has been found that only a very simple aging process is needed to obtain peak strength. Artificial aging at 150°C is not necessary. The material need only be aged at room temperature for a time of 7 hours or more. Thus industry should be able to heat treat the rolled composite material much more efficiently and inexpensively than a T6 heat treatment would permit.

The large precipitates weaken the composite in three basic ways:

1) The growth of large precipitates can weaken the material by removing the small precipitates which are needed to help prevent dislocation motion, thus slip. Therefore, the shear strength of the matrix is reduced which results in the need for a larger critical whisker length. The existing shorter whiskers are unable to carry as much load and the material is weakened.

2) The large precipitates tend to create large voids in the composite which then act as crack initiation sites and also provide low energy crack paths.

3) Evidence shows that the precipitates tend to nucleate at dislocations near the interface. Therefore, the precipitates tend to grow around the whiskers and envelope them, thus minimizing the strengthening effect of the whiskers.

It was shown that the Al₂124 matrix provided a much stronger composite than a pure aluminum matrix, at least up to a temperature of 400°C. At temperatures higher than this it appears as if precipitation strengthening offers no contribution to the strength. It has also been shown that the reinforcement itself offers little contribution to the strength at temperatures above 300°C, due to the very low shear strengths in the matrix at these high temperatures. Elevated temperature shear tests showed that the main reason for the drop in tensile strength at higher temperatures was due to a rapid decrease in shear strength of the matrix.

It was also found that not only does the T6 heat treatment increase the strength of the rolled composite but it also in-

creases the room temperature ductility. The AR material has very poor ductility because the large precipitates initiate cracks, which lead to tensile fracture before much plastic deformation can take place. However, the ductility of the AR material increases with temperature to a maximum near 350°C, while the ductility of the T6 material decreases to a minimum at a temperature of 300°C. The precipitates are also thought to be responsible for these effects.

The extruded Al2124 15% SiCw composite was much less homogeneous than the hot rolled material. Precipitates were observed to grow in linear groups along the extruded direction apparently due to variations in plastic deformation.

This program has generated a total of eleven scientific papers and/or presentations. Also Master of Science degrees were granted to C.R. Harris, who is presently in the industrial work force, and R.D. Schueller, who is continuing his studies toward the Ph.D. degree at the University of Virginia.

APPENDIX

ACCEPTED FOR PUBLICATION IN JOURNAL OF
COMPOSITE MATERIALS (IN PRESS, 1990)

THE EFFECTS OF HOT ROLLING ON AA2124 15 v\o
SiC WHISKER COMPOSITES

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ABSTRACT

Mechanical and microstructural studies were performed on a rolled sheet of AA2124 15 volume percent SiC whisker composite. Tests were done in both the as-rolled condition and after a T6 heat treatment. It was confirmed that hot rolling significantly reduces the tensile strength of a heat treated composite and that a T6 heat treatment restores this strength. It has been suggested by previous investigators that this reduction and recovery in tensile strength was due to the creation and healing of interfacial voids. However, the present study shows that the loss in strength is mainly due to simple overaging of the rolled composite which occurs during cooling. A T6 heat treatment restored the strength of the rolled composite by dissolving the large precipitates. The ductility of the composite was also increased significantly as a result of the heat treatment.

1. INTRODUCTION

Aluminum matrix composites are known to have the beneficial properties of high strength and high modulus combined with the ability to withstand higher temperatures than unreinforced aluminum. However, in order to take advantage of these properties methods must be found to process the aluminum matrix composites into the various shapes and sizes necessary for use. The present research focuses on the processing step of hot rolling and its effect on AA2124-SiC whisker composites.

It was noted in earlier papers, by Harris et al. (1) and by Harrigan et al. (2) that when a heat treated AA2124 composite with SiC reinforcement was hot rolled it lost a large percentage of its tensile strength. It was found that when this composite was subsequently heat treated in the T6 condition its strength was restored. Harrigan et al. (2) gave no explanation for this effect, whereas, Harris et al. (1) proposed that the strength decrease from hot rolling was a result of void formation or cavitation at the whisker-matrix interface. The subsequent heat treatment was then thought to heal these interfacial voids, thus restoring the strength. This conclusion was based on observations from AA1100/SiC whisker composites (a non heat treatable alloy matrix) and were generalized to other alloy systems. The present study has found that in a precipitation hardenable matrix, such as AA2124, other mechanisms are responsible for the loss in tensile strength

due to hot rolling. The investigation of these mechanisms is the focus of the present study.

2. EXPERIMENTAL PROCEDURE

2.1 Material

A rolled sheet AA2124 (SXA24E) reinforced with 15 volume percent SiC whiskers was received from Advanced Composites Materials Corporation in Greer, SC. This sheet was rolled to a thickness of 1.8 mm (0.071 inches); approximately an 80 percent reduction. The rolling was done at a temperature of 438°C with an 8% reduction per pass. Between the passes the material was solutionized for an hour at 495°C in order to prevent fracture. The last few passes were done at room temperature.

2.2 Tensile Tests

Tensile bars of length 7.5 cm were cut from the rolled material. The center 5 cm of these bars were rounded on the lathe with 600 grit paper. These samples were tested using an Instron Tensile Testing Machine at an initial strain rate of 0.00067 sec⁻¹. The samples were tested in the as-rolled condition and after a T6 heat treatment (1 hour at 500°C, cold water quench, and then 8 hours of artificial aging at 150°C).

Tensile strain to failure was calculated using the gauge length, the chart speed, and the crosshead speed. The effects of the Instron machine were subtracted away using the method described in Mechanical Metallurgy (3).

2.3 Microstructural Studies

When the cross section of metallographic samples were prepared using conventional polishing methods, it was discovered that the voids, which formed in the material during tensile testing, were filled with debris from the polishing process. Therefore, these voids, as well as other features, could not be seen clearly with the scanning electron microscope (SEM). In order to get a clean smooth surface, the samples were ion milled using a Model 600 Gatan Dual Ion Mill with an argon ion beam. The samples were ion milled for only 3 hours to prevent excessive heating. The beam was set at an angle of 15 degrees for the first two hours and then reduced to 10 degrees for the remaining hour. The voltage was set at 4 keV and the current was set at 0.5 mA to erode the aluminum matrix and the SiC whiskers simultaneously. Thus, a very clean new surface is exposed in which whiskers, voids, precipitates, and in some instances, even grain boundaries can be seen clearly on the SEM.

Transmission electron microscope (TEM) samples were prepared using jet electropolishing with methanol:nitric acid solution in a 3:1 ratio at a temperature of -30°C and a current of 1.2 mA. The specimens were analyzed using a Philips EM 400 TEM with a double tilt specimen holder at an accelerating voltage of 120 kV.

2.4 Shear Tests

Shear tests were made using the punch-and-die

(blanking) method as described in the Metals Handbook (4). The sample was sandwiched between two steel plates with aligned holes of diameter 0.508 cm. A round, flat punch of diameter 0.452 cm was then lowered with the Instron at a crosshead velocity of 0.05 cm/min through the hole in the steel plates. The sample and steel plates were placed on a compression cell so that a shear stress strain curve could be obtained.

2.5 Three Point Bend Studies

To simulate crack growth in a tensile loaded specimen, three point bend tests were performed on material that had been tensile tested. The longer halves of fractured tensile specimens were cut to a length of 3 cm and two notches were cut in them 1 cm from each end. The Instron was used to slowly apply a force (crosshead velocity of 0.005 cm/min) to the center of the sample. When a crack was observed to form at one of the notches, the instron was immediately stopped and the sample removed. The cross section was then polished and ion milled in the manner previously mentioned.

3. RESULTS

3.1 Tensile Tests

Table 1 shows the results of the room temperature tensile tests performed in the longitudinal and transverse orientations. These test results are combined with the results of Harris et al. (1), Harrigan et al. (2) and Birt et al. (5). These tests confirmed that hot rolling does

indeed reduce the tensile strength of heat treated SiCw reinforced aluminum alloys, and that a subsequent T6 heat treatment does restore the strength. Yield strength effects could not be compared since they were affected by the varying degrees of work hardening which occurred as a result of specific rolling conditions.

3.2 SEM Studies

Sample cross sections, prepared in the manner mentioned earlier, were studied on the SEM using back scattered electron imaging (BEI) in the compositional mode. The compositional mode depicts heavier elements with lighter contrast. The precipitates can be seen in Figure 1 as bright clumps in the matrix and are relatively large with a diameter from 1-10 μm . X-ray analysis identified the precipitates as the θ phase, Al_2Cu . These large precipitates were not observed in the material which had undergone a T6 heat treatment.

3.3 Cooling Studies

To show that the precipitates grew as the material cooled down after the solutionizing and hot rolling step, the as-rolled material was solutionized at 500°C for two hours to ensure full dispersion of the precipitates, then allowed to cool steadily from 500°C to 150°C for different periods of time. Figure 2 is a plot of tensile and 0.2% yield strength vs. cooling time for this material. At least three samples were tensile tested in each condition and the error bars show the range of strengths received (when none

are shown the scatter is smaller than the point marker). It is evident that the tensile and yield strength of the material drops off rather steeply with cooling time. In fact the tensile strength seems to converge to a minimum strength near the as-rolled tensile strength. If the material is allowed to cool for 1 hour (a cooling rate of 0.097°C/s), the tensile strength drops to 445 MPa (64.5 ksi) which is essentially the same as the as-rolled strength of 441 MPa (63.9 ksi).

The yield strength of the slow cooled material could not be compared to that of the as-rolled material since the as-rolled material was significantly work hardened as a result of the rolling, whereas the slow cooled material had been annealed.

SEM studies showed that the precipitates did grow back as the material slowly cooled. Figures 3a, b, c, and d are identical magnification BEI compositional photomicrographs of materials that had been cooled for 5, 11, 20, and 60 minutes, respectively. One can see that as expected the precipitates started out rather small and continued to grow larger with increased cooling time. After 60 minutes of cooling, the size of the precipitates was similar to that observed in the as-rolled material.

3.4 Effects of Matrix Overaging

It is evident that when the precipitation hardenable matrix is grossly overaged, as in the as-rolled condition, only large precipitates exist and most of the copper is

incorporated in them. Thus, one is clearly left with an almost pure aluminum matrix which is quite ductile and has a relatively low shear strength (6). However, when the composite material is solutionized and peak aged, many small, closely spaced, precipitates form and dislocation motion is more difficult. Therefore, the matrix shear strength will be greater in the T6 material.

Room temperature shear tests were performed on the material to compare the ultimate matrix shear strengths under the different conditions. These results are given in Table 2 and can further be compared to blanking shear data for AA2024 and AA1100 which were given in the Metals Handbook (4). It is noticed that the shear strength of the T6 material is almost double that of the as-rolled material.

The matrix shear strength is a very important factor in the strength of a composite, since it is the shear strength of the matrix that determines how well the matrix can transfer load to the reinforcement, assuming a strong interface. With the strong interface assumption, the interfacial shear strength is presumed equal to the matrix shear strength, thus the equation for the critical length to diameter ratio is given by Eq. (1),

$$\frac{l_c}{d} = \frac{\sigma_{fmax}}{2\tau_m} \quad \text{Eq. (1)}$$

where σ_{fmax} is the maximum tensile strength of the fiber and τ_m is the matrix shear strength (7). If $l/d < l_c/d$ then the maximum stress transferred to a fiber can be approximated by

rearranging Eq. (1) to give Eq. (2),

$$\sigma_f = 2\tau_m \frac{1}{d} \quad \text{Eq. (2)}$$

where σ_f is the maximum stress that can be transferred to the fiber. Therefore, when the as-rolled material is heat treated, the increase in matrix shear strength allows the shorter whiskers to carry more load resulting in a stronger composite, as was observed in this study.

3.5 Crack Initiation Sites and Low Energy Crack Paths

Another way in which the large precipitates lower the tensile strength of the material is by creating large voids which can act as crack initiation sites or provide a low energy crack path. Upon loading, nearly all the relatively large (5-10 μm) Al_2Cu precipitates fracture prematurely, leaving large voids between the halves, as can be seen in Figure 4a, a compositional mode photomicrograph of the longitudinal section below the fracture surface of as-rolled material tensile tested at room temperature. Fewer of the smaller (1-5 μm) precipitates fracture upon loading. Figure 4b shows a crack which is initiating at one of the large fractured precipitates. Thus, when stress is applied to this composite material, the large voids caused by particle fracture lead to easy crack initiation, growth, and subsequent composite fracture.

As mentioned earlier, the crack path in a tensile loaded specimen was simulated by initiating a crack in previously tensile tested specimens using the three point

bend method. The crack in the as-rolled material was seen to advance through the formerly fractured precipitates, as shown in Figure 5a. In the T6 material, however, there were no large precipitates and the crack tip instead advanced mainly through the small voids which previously grew at the whisker ends, as can be seen in Figures 5b and 5c. Initially this observation appears to contradict work by Lewandowski et al. (8), Liu et al. (9), and Shang et al. (10) which propose that cracks grow mainly through SiC particles in an underaged material with a fine distribution of reinforcement. However, the cracks in those studies were initiated in untested material, whereas in the present study, cracks were initiated in prestressed material which subsequently had preexisting voids at the whisker ends.

3.6 Precipitate Coarsening Adjacent to Whiskers

The third way in which the large precipitates lowered the composite strength is by nucleating and growing at the whisker interface. To observe the nucleation of the precipitates, the cross sections of slowly cooled samples were studied. The precipitates in these samples were smaller than in the as-rolled material and in earlier stages of growth. Looking back at Figure 3b it can be seen that many of the precipitates are nucleating on a whisker. TEM studies have confirmed this observation; many instances were found in which precipitates nucleated and grew on whisker interfaces (see Figures 6a and 6b). Others such as Nutt (11), Xiu-Qin et al. (12), and Kohyama et al. (13), have

also found whiskers to be preferential sites for precipitate nucleation. Nutt and Phillips (14) have shown the whiskers to have many small facets on their surface. These facets may be the nucleation sites.

It can further be seen in Figure 7a that as precipitates grow, they begin to cover the interface of the whisker until the whisker is completely enveloped. The cross section of an as-rolled sample observed parallel to the whiskers shows the advanced stages of precipitate growth (see Figure 7b). One can see that each precipitate tends to cover several whiskers, thus a large number of whiskers are affected.

When the short whiskers are engulfed in brittle Al_2Cu not much load can be transferred to them because the whisker and the Al_2Cu are both brittle materials. This explains why many precipitates with whiskers pulled out of them were noticed on the fracture surfaces. Figure 8 shows an example of how the whiskers simply pull out of the precipitates and are thus unable to help strengthen the material.

3.7 Effects of Hot Rolling on Ductility

It can be seen from Table 2 that the room temperature strain to failure of the T6 material is almost triple that of the as-rolled material in both the longitudinal and the transverse directions. The stress strain curves showed that the as-rolled material underwent very little plastic deformation (only about 0.8% in the longitudinal orientation), while the T6 material plastically deformed

much more (approximately 2.5%).

4. DISCUSSION

In Figure 2 it was observed that the tensile strength of the solutionized and slow cooled material approached that of the as-rolled material. This fact suggests that interfacial damage in the form of cavitation is not a significant factor in the decline of tensile strength after hot rolling in the AA2124-SiCw system. Interfacial voids would have healed during the solutionizing step, thus the following cooling period would not have an adverse effect on the strength. Therefore, the tensile strength would not decline and approach the as-rolled tensile strength upon slow cooling if the formation and healing of interfacial voids were involved.

Thus cavitation at the interface is not thought to be the main cause of strength loss in the AA2124-SiCw system. Instead it has been found in this study that the reduced tensile strength is due to simple overaging of the precipitation hardenable matrix. During fabrication the composite material was solutionized, then hot rolled at temperatures of 438°C, and then allowed to cool in air. The furnace cooling experiments showed that the overaging most likely took place during this cooling period.

A direct comparison to unreinforced 2024 further supports the fact that simple matrix effects are responsible for the decline in strength of the composite after rolling.

After solutionizing at 500°C, cold water quenching, and natural aging the composite and the 2024 had UTS values of 687 MPa (99.7 ksi) and 469 MPa (68 ksi), respectively (6). However if a cooling rate of 1.5 °C/s was introduced the tensile strengths dropped to 494 MPa (71.7 ksi) and 379 MPa (55 ksi) (15), respectively. Thus, the drop in tensile strength upon slow cooling was larger in the composite, as would be expected if matrix overaging was responsible for the loss in strength of the composite.

It was noted earlier that the as-rolled material underwent very little plastic deformation. This lack of plastic deformation may be due to two factors. Firstly, the as-rolled material was likely work hardened somewhat during the rolling process which would raise the yield strength and lower the plastic deformation. Secondly, as described earlier, the large precipitates in the matrix act as crack initiation sites. Thus cracks form and the composite fractures prematurely before much deformation can take place. When the material is solutionized and cold water quenched the large precipitates are dissolved and many of the dislocations are annealed out.

The strain to failure of the slow cooled material could not be compared to that of the as-rolled material since the cooling rate experiments only take into account the effect of the precipitates while the as-rolled material also includes the effect of work hardening from the rolling process.

5. CONCLUSIONS

It has been confirmed that when a peak aged AA2124 15v\o SiCw composite was hot rolled and allowed to slowly cool, it loses a good portion of its tensile strength. And when this composite was subsequently subjected to a T6 heat treatment the strength was regained. However, it was shown that the loss in strength was mainly due to simple overaging of the precipitation hardenable matrix and not from the growth of interfacial voids. Large Al_2Cu precipitates grew as the material was allowed to cool after the hot rolling step. The strength was regained mainly because the heat treatment dissolved the coarse precipitates and provided a more homogeneous microstructure.

The precipitates were found to weaken the composite in three basic ways:

(1) The shear strength of the matrix declined which reduced the load carried by the whiskers.

(2) The large precipitates also tended to prematurely fracture, thus creating large voids in the composite which acted as crack initiation sites and also provided low energy crack paths.

(3) Finally, evidence showed that the precipitates tended to nucleate and grow near the interface of the whiskers. Therefore, the precipitates enveloped many of the whiskers, thus preventing them from carrying much load.

It was also discovered that not only did a heat treatment increase the tensile strength of the hot rolled

AA2124/SiCw composite but it also significantly increased the ductility.

Acknowledgments

The authors appreciate the support of the Office of Naval Research, Dr. Steven G. Fishman, Scientific Officer, under grant #N00014-85-K0179.

References

1. C.R. Harris and F.E. Wawner, Processing and Properties for Powder Metallurgy Composites, pp. 107-115, The Metallurgical Society, 1988.
2. W.C. Harrigan, Jr., G. Gaebler, E. Davis and E.J. Levin, Mechanical Behavior of Metal Matrix Composites, pp. 169-180, Metallurgical Society of AIME, 1982.
3. M. A. Meyers, K.K. Chalwla, Mechanical Metallurgy, p. 562, Prentice Hall Inc., 1984.
4. Metals Handbook, 9th edition, vol. 8, p. 64, Mechanical Testing, American Society for Metals, 1985.
5. M.J. Birt and W.S. Johnson, "Characterization of the Mechanical and Microstructural Properties of an Aluminum MMC", Presented at TMS Fall Meeting, Indianapolis, IN, 1989.
6. Aluminum: Properties and Physical Metallurgy, Ed. J. Hatch, American Soc. for Metals, Metals Park, Ohio, p. 358, 362, 1984.
7. A. Kelly and G.J. Davies, J. Metallurgical Reviews, 10, pp. 25-26, No. 37, 1965.
8. J.J. Lewandowski and C. Liu, Processing and Properties for Powder Metallurgy Composites, Ed. P. Kumar, K. Vedula and A. Ritter, The Metallurgical Society, pp. 117-137, 1988.
9. C. Liu, S. Pape, and J.J. Lewandowski, Interfaces in Polymer, Ceramic, and Metal Matrix Composites, Ed. H. Ishida, Elsevier, New York, pp. 513-524, 1988.
10. J.K. Shang, L.H. Edelson, J.J. Mason, W. Yu, and R.O. Ritchie, Interfaces in Polymer, Ceramic, and Metal Matrix Composites, Ed. H. Ishida, Elsevier, New York, p. 703, 1988.
11. S.R. Nutt, Interfaces in Metal-Matrix Composites, Ed. A.K. Dhingra and S.G. Fishman, pp. 157-167, The Metallurgical Society, 1986.
12. Chen Xiu-Qin and Hu Geng-Xiang, Interfaces in Polymer, Ceramic, and Metal Matrix Composites, pp. 381-389, Elsevier, 1988.
13. Akira Kohyama, Hideshi Tezuka and Shuntaro Saito, Interfaces in Polymer, Ceramic, and Metal Matrix Composites, pp. 125-139, Elsevier, 1988.

14. S. R. Nutt and D.S. Phillips, Interfaces in Metal-Matrix Composites, pp. 111-120, The Metallurgical Society, 1986.

15. Metals Handbook, p. 698, American Society for Metals, 9th Edition, Vol. 4., 1985.

TABLE 1. Ultimate tensile strengths of aluminum composites.

| <u>MATERIAL</u> | <u>Extruded (T6)</u> | <u>HOT ROLLED</u> | |
|--|----------------------|-------------------|--------------------|
| | | <u>As-Rolled</u> | <u>Rolled (T6)</u> |
| 2124-20v/o SiC _w [1] | 700 | 430 | 673 |
| 6061-15v/o SiC _w [2] | 443 | 258 | 454 |
| 6061-30v/o SiC _w [2] | 498 | 270 | 501 |
| 2124-15v/o SiC _w (SXA24E) Longitudinal | 670 [5] | 447 | 678 |
| Transverse | * | 353 | 557 |

* Data for transverse extruded SXA24E-15v/o SiC_w was not available.

TABLE 2. Ultimate shear strength and tensile strain to failure for rolled AA2124 15v/o SiCw and other alloys.

| Condition | ULTIMATE SHEAR STRENGTH | | TENSILE STRAIN TO FAILURE |
|----------------------------|-------------------------|-------|---------------------------|
| | (MPa) | (Ksi) | % |
| As Received Transverse | 181 | 26.3 | 3.2 |
| T6 Transverse | 319 | 46.3 | 9.6 |
| As Received Longitudinal | -- | -- | 1.3 |
| T6 Longitudinal | -- | -- | 3.4 |
| T6 AA2014 | 284 | 41.2 | -- |
| AA1100 (commercially pure) | 66 | 9.6 | -- |

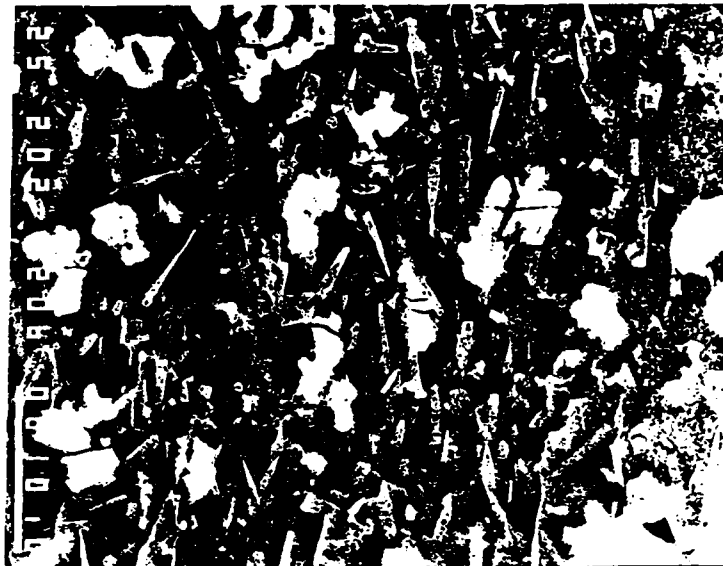


Figure 1. SEM compositional micrograph of as-rolled 2124 15v/o SiCw composite.

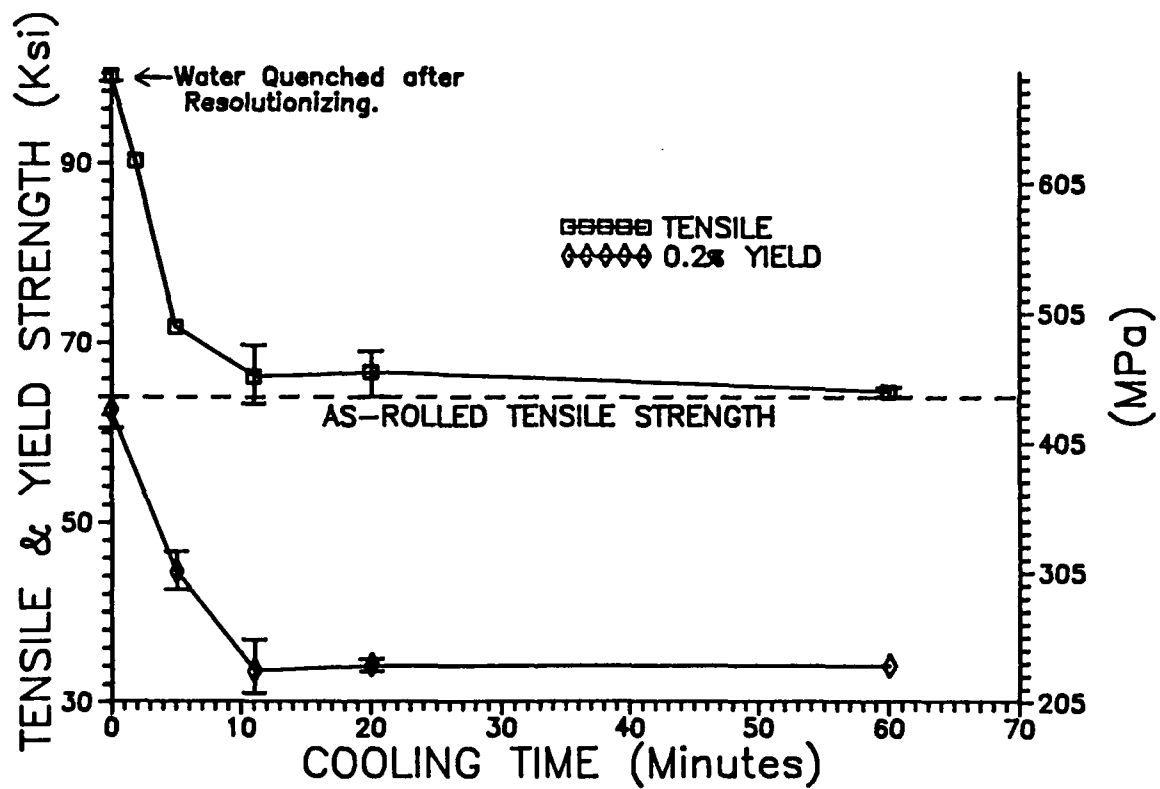


Figure 2. Cooling time was the time to cool from 500°C to 150°C.



(a)



(b)

10 μm

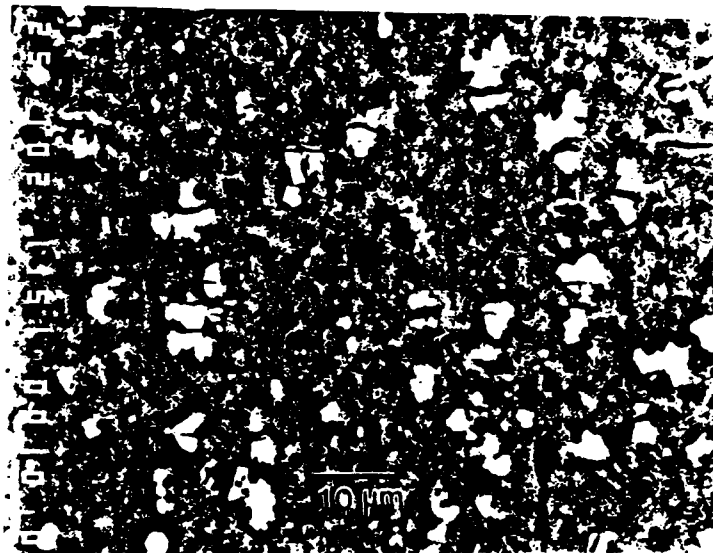


(c)

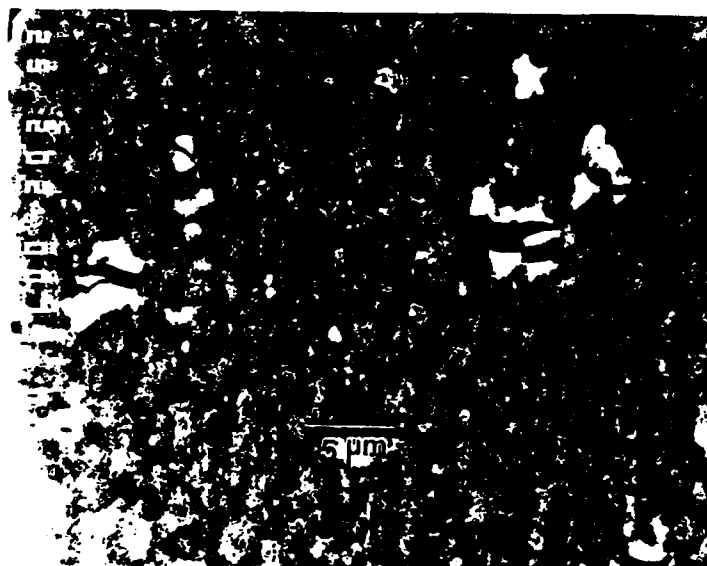


(d)

Figure 3. AA2124 15v/o SiCw composite material which was solutionized and then allowed to furnace cool from 500°C to 150°C for different periods of time, a: 5 minutes, b: 11 minutes, c: 20 minutes, d: 60 minutes.

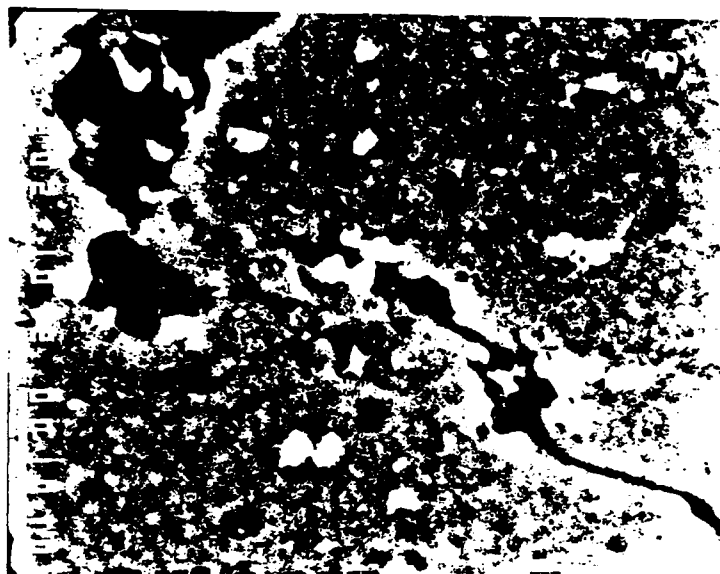


(a)



(b)

Figure 4. The cross section of as-rolled material which had been tensile tested at room temperature, a: many of the large precipitates fracture upon loading, b: a crack initiating at a fractured precipitate.



(a)



(b)



(c)

Figure 5. The cross section of cracks created by three point bending, a: crack in as-rolled material, b,c: the crack tip in T6 material instead going through the voids created at the ends of whiskers.

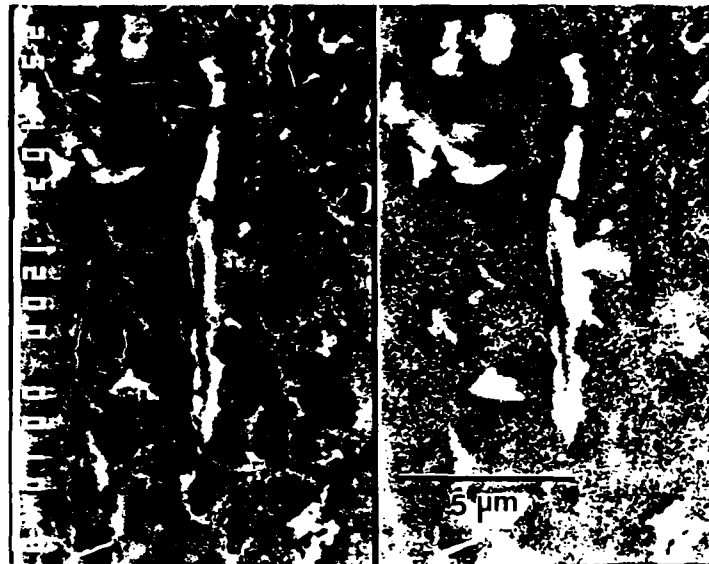


(a)

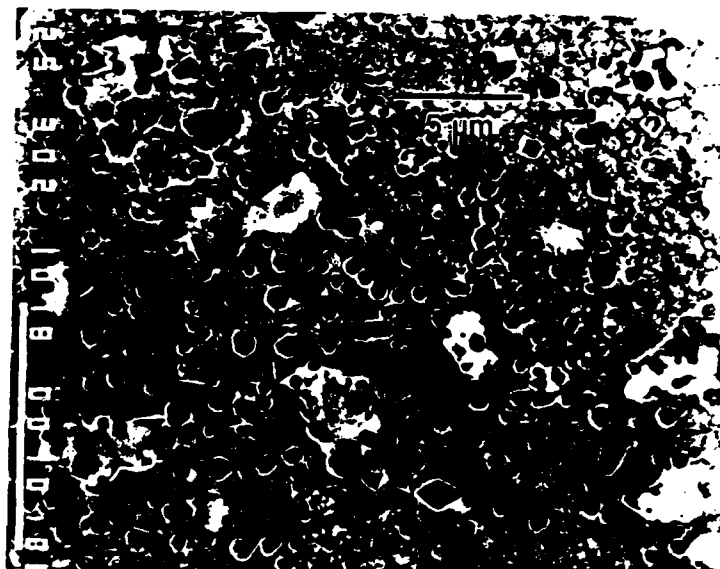


(b)

Figure 6. TEM of precipitates nucleating on the shisker interface.



(a)



(b)

Figure 7. SEM a: slow cooled material in which a precipitate has completely engulf a single whisker, b: as-rolled material observed parallel to whiskers showing how each precipitate engulfs several whiskers at once.



Figure 8. Precipitate on fracture surface of as-rolled material. The whiskers simply pullout of the fractured precipitate.

ACCEPTED FOR PUBLICATION BY COMPOSITES
SCIENCE AND TECHNOLOGY (IN PRESS 1990)

AN ANALYSIS OF HIGH TEMPERATURE BEHAVIOR OF AA2124 SiC WHISKER COMPOSITES

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ABSTRACT

AA2124 SiC whisker composites were received from a commercial source in the hot rolled and extruded condition. Tensile and shear tests were carried out on these materials at various temperatures in order to develop a better understanding of the failure mechanisms involved at high temperatures. It was discovered that the loss in strength of aluminum composites with increasing temperature was mainly due to a decrease in matrix shear strength. Precipitation kinetics contribute to this loss in shear strength through the resolution of precipitates at higher temperatures. This information is necessary in order to develop aluminum base composites with high temperature capabilities.

1. INTRODUCTION

It has been recognized for some time that understanding microstructural and fracture relationships is essential to producing a high temperature aluminum composite material of high and consistent quality. Merely adding a strong reinforcing phase to a good aluminum alloy does not necessarily guarantee a

better high temperature material. Much information can be gained by determining what effect elevated temperature has on composite properties such as tensile strength, shear strength, ductility, and fracture. This knowledge can help in making aluminum composites for higher temperature applications. These composites would be very useful in replacing, for example, the much denser titanium alloys used in mid-temperature range aerospace applications.

Upon evaluating the literature on various discontinuously reinforced aluminum alloys, many data were found relating tensile strength to temperature [ref 1-5]. Figure 1 is the plot of this data for aluminum composites with five different matrices, two different reinforcements, four different fabrication techniques and two different fiber orientations. It can be seen that, although there are vastly different room temperature properties, the strength values drop off rather quickly above 300°C and converge to a strength of about 70 MPa. Thus the objective of this research was to determine what microstructural aspects of the composite are responsible for the major drop in strength near and above 300°C.

2. EXPERIMENTAL PROCEDURE

2.1 Material

A rolled sheet of AA2124 (SXA24E, with 4% Cu, 1.4% Mg, and 0.2% Fe) reinforced with 15 volume percent (15v/o) SiC whiskers was received from Advanced Composites Materials Corporation (ACMC) in Greer, SC. This sheet was rolled to a thickness of 1.8 mm (0.071 inches); approximately an 80 percent reduction.

Also received from ACMC was an extruded plate of AA2124 (SXA24E) reinforced with 20 v\o SiCw. The extrusion conditions were not disclosed by the manufacturer.

2.2 Tensile Tests

Tensile specimens were made by cutting 7.5 cm bars and rounding the center 5 cm on the lathe with 600 grit paper. Samples were cut in both the longitudinal and transverse directions and were tested using an Instron Machine at an initial strain rate of 0.00067 sec^{-1} . The material was tested in both the as-rolled (AR) condition and after a T6 heat treatment (500°C for 1 hour, cold water quench, artificially aged 150°C for 8 hours). These tensile tests were conducted at various temperatures ranging from room temperature to 450°C . Specimens were heated using a resistance tube furnace. A thermocouple was attached to the specimen and the furnace was allowed to heat for about 15 minutes until the desired temperature was reached. The sample was then held at that temperature for another 15 minutes to ensure equilibrium. At least three samples were tested at each condition.

2.3 Shear Tests

Shear tests were made using the punch-and-die method (blanking) described in the Metals Handbook [6]. The sample was sandwiched between two steel plates with aligned holes of diameter 0.508 cm. A round, flat punch of diameter 0.452 cm was then lowered with the Instron at a rate of 0.05 cm/min through the hole in the steel plates until a piece was punched out of

the specimen. The sample and steel plates were placed on a compression cell so that a shear stress strain curve could be obtained. This setup could also be placed in a resistance furnace so that high temperature shear strengths could also be determined. Ideally the punch should be aligned along the whisker direction to get accurate results, however this was not possible since thin plate material was used. However, since the whiskers are fairly short (lengths of about $5\mu\text{m}$) small deviations in matrix shear strength are expected. One should also keep in mind that the values obtained are only used to get temperature trends and are not used to generate a data base.

2.4 Microstructural Studies

The cross section of metallographic samples were prepared using conventional polishing methods followed by a short period of ion milling (about 3 hours) at a voltage of 4 kev and a current of 0.5 mA. With this method, a clean new surface is exposed in which whiskers, voids, precipitates, and in some instances even grain boundaries could be seen clearly on the SEM.

3. RESULTS AND DISCUSSION

3.1 Effects of Temperature on Strength

Figure 2 shows the results of the hot rolled AA2124 15v/o SiCw composite tensile tests at elevated temperatures. It is apparent that at ambient temperatures the tensile strength of the as-rolled material is much lower than that of the T6 material. Earlier work has shown this reduced tensile strength to be a result of matrix overaging which occurs as the material

slowly cools from the hot rolling temperature [7]. This effect has also been observed in other aluminum alloy composites as can be seen in Table 1 [8-10].

Also included in Figure 2 are data for extruded AA1100 20v/o SiCw composite. It can be seen that these composites behave similarly to those shown in Figure 1. The tensile strengths drop off at about 300°C and converge to a value near 70 MPa. At temperatures over 350°C the strengths of the AA1100 and the AA2124 composites are nearly identical, implying that the precipitates become dissolved in the AA2124 composite and offer no contribution to the strength. It was also observed that the orientation of the reinforcement does not seem to have any influence on tensile strength at temperatures above 350°C. Thus it seems the reinforcement itself does not contribute to the strength at these higher temperatures.

Figure 3 shows tensile data for extruded AA2124 20v/o SiCw and for unreinforced extruded AA2124 both prepared by the same powder metallurgy fabrication procedure and subjected to a T6 heat treatment. The composite has considerably higher strength up to a temperature of 300°C. At and above this temperature the strengths are nearly identical, implying that in this case the whiskers basically carry no load at temperatures above 300°C. The drop in strength in the extruded composite occurs at a lower temperature than in the hot rolled composite possibly due to the more homogeneous microstructure obtained from hot rolling [11].

It is believed that as the temperature is raised, the tensile strength of the composite declines mainly due to the

inability of the matrix to transfer load to the whiskers. The critical factor which determines how well the matrix can transfer load to the reinforcement, assuming a strong interface, is the matrix shear strength. This relationship is recognized in the equation for the critical length to diameter ratio, which describes the value necessary to load a fiber to its ultimate strength. The equation for this ratio is given as

$$\frac{l_c}{d} = \frac{\sigma_{fmax}}{2\tau_m} \quad \text{Eq. 1.}$$

where σ_{fmax} is the ultimate tensile strength of the fiber and τ_m is the matrix shear strength [12]. If $l/d < l_c/d$ then the maximum stress transferred to a fiber can be written,

$$\sigma_f = 2\tau_m \frac{l}{d} \quad \text{Eq. 2.}$$

Thus, as the matrix shear strength is reduced, less load is transferred to the fibers resulting in a lower composite tensile strength.

Figure 4 shows the matrix shear strength of rolled composite material at increasing temperatures. It can be seen that the shear strength of the as-rolled and T6 materials decline significantly and converge to similar values of about 30 MPa at temperatures above 300°C

Low matrix shear strength is also evident upon examination of the fracture surfaces of samples tested at high temperature. Figure 5 is a fracture surface of a sample tested at 450°C, showing a non-dimpled appearance, intergranular fracture, and exposed whiskers. In some instances the matrix has even sheared

away from whiskers with aspect ratios greater than 20.

The decrease in shear strength with increasing temperature can also be seen by observing the size of voids at the ends of whiskers inside tensile tested material. Figure 6 consists of micrographs of the cross sections of rolled AA2124 15v/o SiCw near the fracture surface. At room temperature the voids are very small and in their beginning stages (Figure 6a). They nucleate at the stress concentration sites at the edges of the whiskers as was shown by Nutt [13]. However, when the temperature is raised to 450°C, the voids become much larger, sometimes nearly as long as the whisker itself (Figure 6b). At this elevated temperature, the matrix shear strength is so low that the whiskers are literally pulled through the matrix and contribute negligibly to the strength.

3.2 Effects of Temperature on Strain

Figure 7 is a graph of engineering strain to failure as a function of temperature for the as-rolled and T6 hot rolled composites tested in the transverse and longitudinal orientations. It can be seen that the strain to failure in the transverse direction is generally much higher than that in the longitudinal direction, as is expected since the whiskers constrain the material much more in the longitudinal direction. However, at temperatures near 450°C the whiskers no longer constrain the matrix, thus the strain is essentially the same for all the samples at these high temperatures.

Most noticeable however, is that the heat treated material reaches a minimum ductility at 300°C while the as-rolled

material reaches a maximum ductility near this temperature. Since the same response was noticed in both the longitudinal and the transverse orientations it is felt that precipitation kinetics are responsible for this effect. One possible explanation is that at temperatures near 300°C the precipitates in the as-rolled material grew even coarser thus leaving behind an even more ductile aluminum matrix. Also the dislocations generated during rolling would be annihilated at temperatures near 300°C . Both of these factors would result in a more ductile material.

However, in the T6 material only small closely spaced precipitates exist. It is possible that near 300°C these precipitates grew slightly larger, thus giving the material a more peak aged condition. This peak aged condition could result in the slightly decreased ductility observed. The ductilities approach similar values at 450°C since the precipitates resolutionize at this high temperature.

4. CONCLUSIONS

The elevated temperature tensile strength of AA2124 SiC whisker composites behaves similarly to other discontinuous fiber reinforced aluminum alloys reported in the literature. It has been observed that at temperatures above 300°C the whiskers contribute negligibly to the composite strength due to the extremely low shear strength of the matrix. It has also been found that the precipitates offer no contribution to the strength above a temperature of 350°C since the precipitates become dissolved in the material.

Thus, the strength values begin to converge at temperatures above 300°C until at 400°C they are all nearly identical. At these high temperatures, the matrix is the controlling feature. A thorough understanding of the mechanism of failure at temperatures above 0.6 T_m is necessary in order to develop aluminum base composites with high temperature capabilities.

The strain to failure in the composite was also greatly affected by temperature increases. However the effects on strain seemed more complex than those on strength. The strain increased to a maximum near 300°C in the AR material while in the T6 material it decreased to a minimum at this temperature. It is thought that precipitation kinetics are somehow responsible for this effect. As occurred with the strength values, the strain values begin to converge at temperatures above 350°C. At 450°C, fiber orientation and precipitation kinetics no longer have an effect on the strain and the values become nearly identical.

Acknowledgements

The authors appreciate the support of the Office of Naval Research, Dr. Steven G. Fishman, Scientific Officer, under grant #N00014-85-K0179.

REFERENCES

1. J. Dinwoodie, E. Moore, C. Langman, W. Symes, Proceedings of the 5th International Conference on Composite Materials, edited W. Harrigan, J. Strife, A. Dhingra, The Metallurgical Society, Warrendale, PA, 1985, p. 671-685.
2. L. Ackermann, J. Charbonnier, G. Desplanches, H. Koslowski, *Ibid.*, p. 687-698.
3. A. Sakomoto, H. Hasegawa, Y. Minoda, *Ibid.*, p. 699-705.
4. A. Sakomoto, H. Hasegawa, Y. Monoda, *Ibid.*, p. 705-707.
5. AA1100 20% SiCw tested by W. Pollock, University of Virginia, Private Communication.
6. Metals Handbook, 9th edition, vol. 8: *Mechanical Testing*, American Society for Metals, Metals Park, Ohio, (1985) p. 64.
7. R.D. Schueller and F.E. Wawner, The Effects of Hot Rolling on AA2124 15% SiCw Composites, submitted to *Journal of Composite Materials*.
8. C. Harris and F. Wawner, Processing and Properties for Powder Metallurgy Composites, TMS Publication, Warrendale, PA, pp. 107-116, 1987.
9. W.C. Harrigan, Jr., G. Baebler, E. Davis and E.J. Levin, Mechanical Behavior of Metal Matrix Composites, ed. J.F. Hack and M.F. Amateau, TMS of AIME, 1982, 169-180.
10. M.J. Birt and W.S. Johnson, presented at TMS Fall Meeting, Indianapolis, IN, 1989.
11. T. G. Nieh and R. F. Karlak, *Journal of Materials Science Letters*, 2 (1983), pp. 119-122.
12. A. Kelly and G. J. Davies, *Metallurgical Reviews*, 10, 37 (1965), pp. 25-26.
13. S. R. Nutt and A. Needleman, *Scripta Met.* 21 (1987), pp. 705-710.

Table 1. Ultimate tensile strength of extruded and hot rolled aluminum matrix composites at room temperature.

| <u>ULTIMATE TENSILE STRENGTH (MPa)</u> | | | |
|--|----------------------|-------------------|--------------------|
| <u>MATERIAL</u> | <u>Extruded (T6)</u> | <u>HOT ROLLED</u> | |
| | | <u>As-Rolled</u> | <u>Rolled (T6)</u> |
| 2124-20v/o SiC _w [1] | 700 | 430 | 673 |
| 6061-15v/o SiC _w [2] | 443 | 258 | 454 |
| 6061-30v/o SiC _w [2] | 498 | 270 | 501 |
| 2124-15v/o SiC _w (SXA24E) Longitudinal | 670 [5] | 447 | 678 |
| Transverse | * | 353 | 557 |

* Data for transverse extruded SXA24E-15v/o SiC_w was not available.

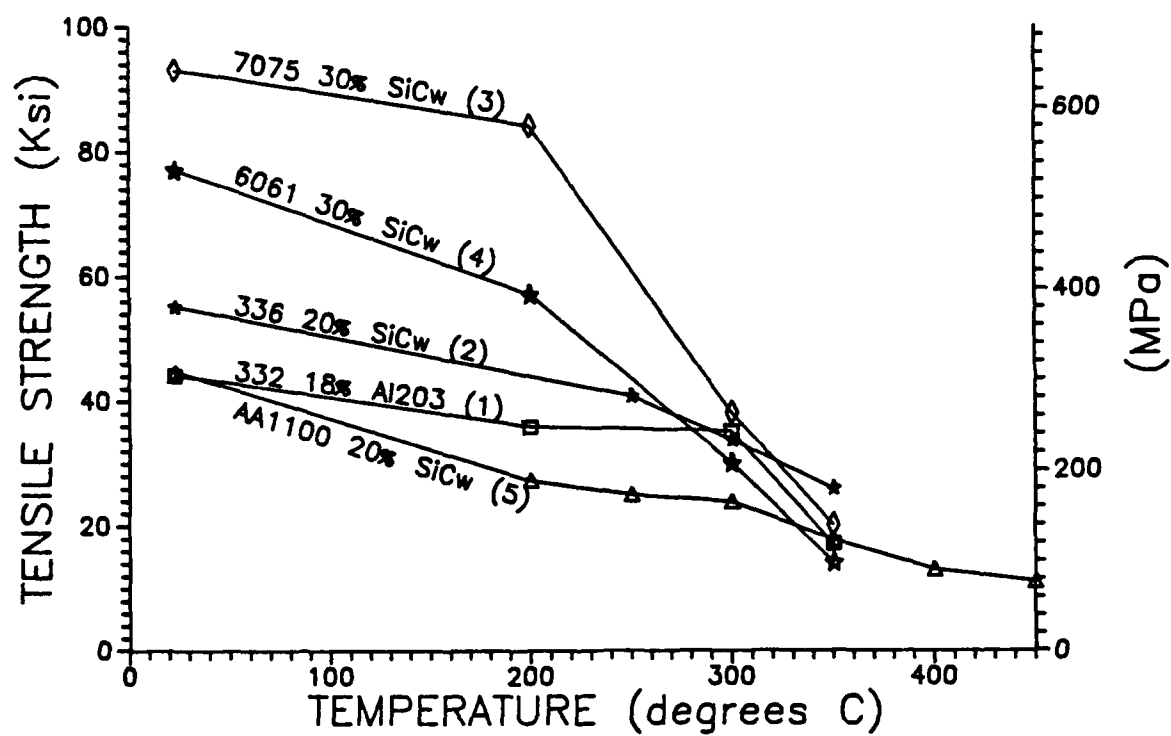


Figure 1. Tensile strength at different temperatures for various aluminum matrix composites.

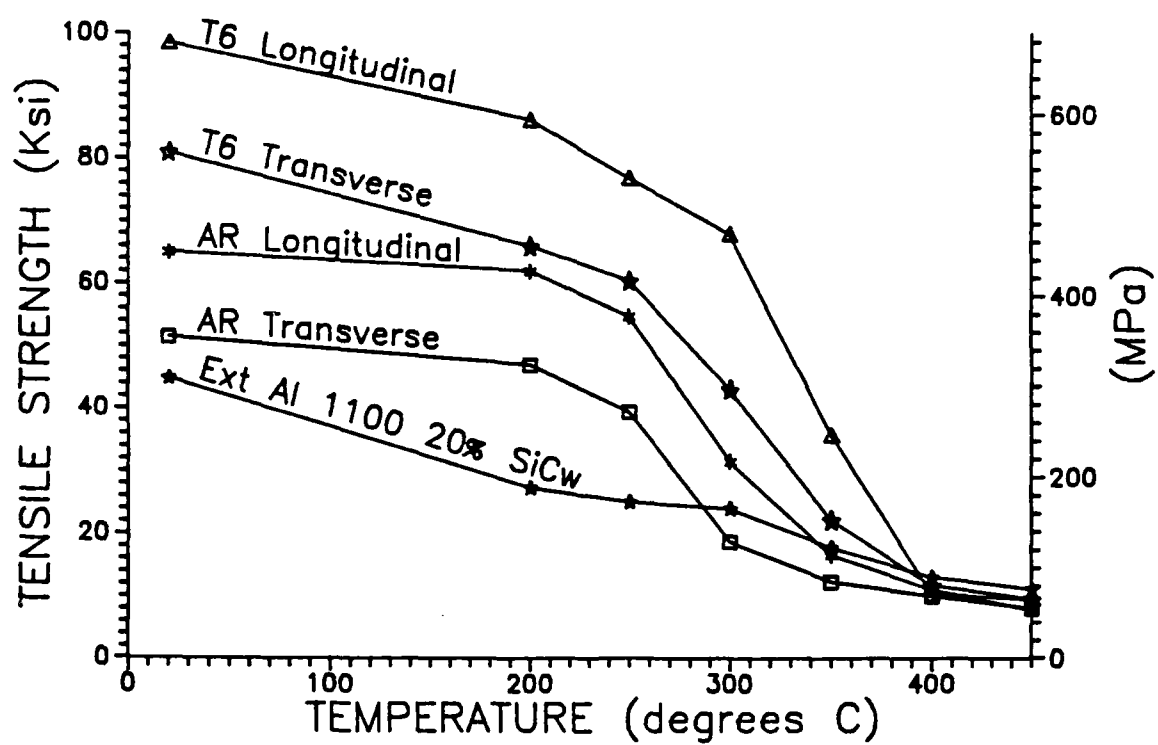


Figure 2. Longitudinal and transverse tensile strengths for hot rolled AA2124 15v/o SiCw composites in the as-rolled and T6 conditions.

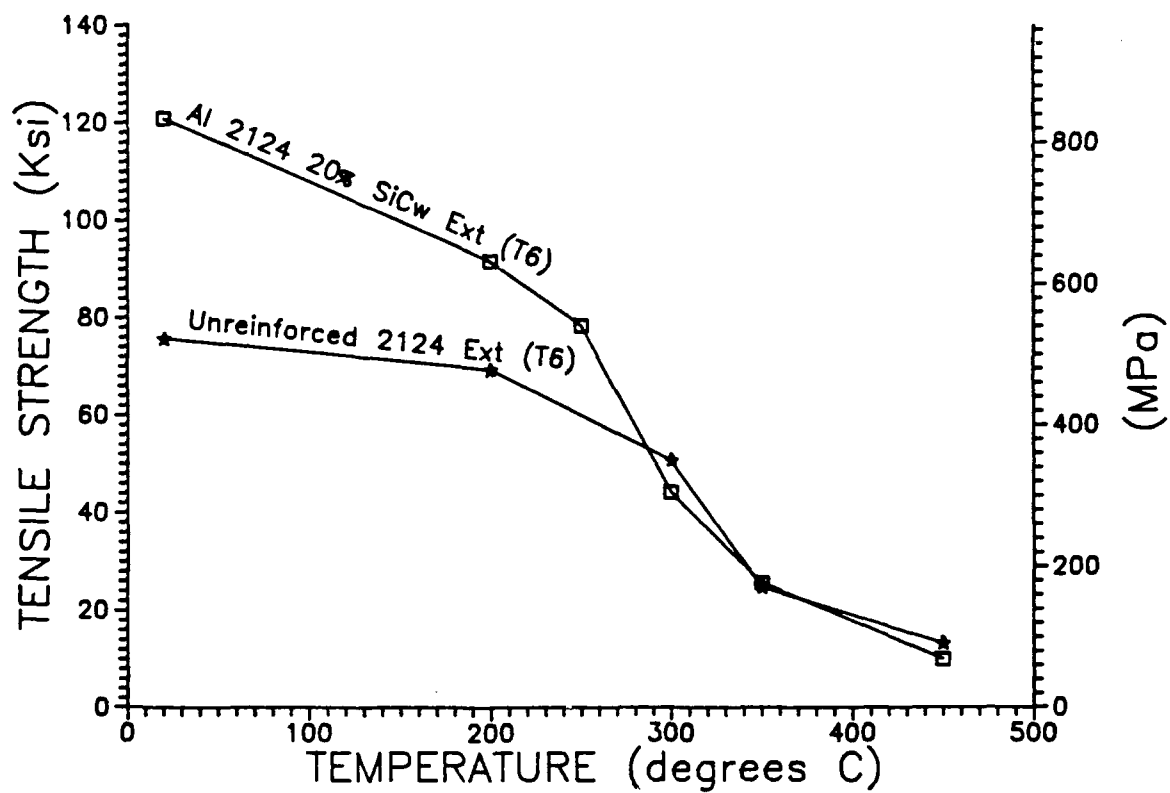


Figure 3. Tensile strengths of unreinforced extruded 2124 and extruded 2124 reinforced with 20v/o SiCw.

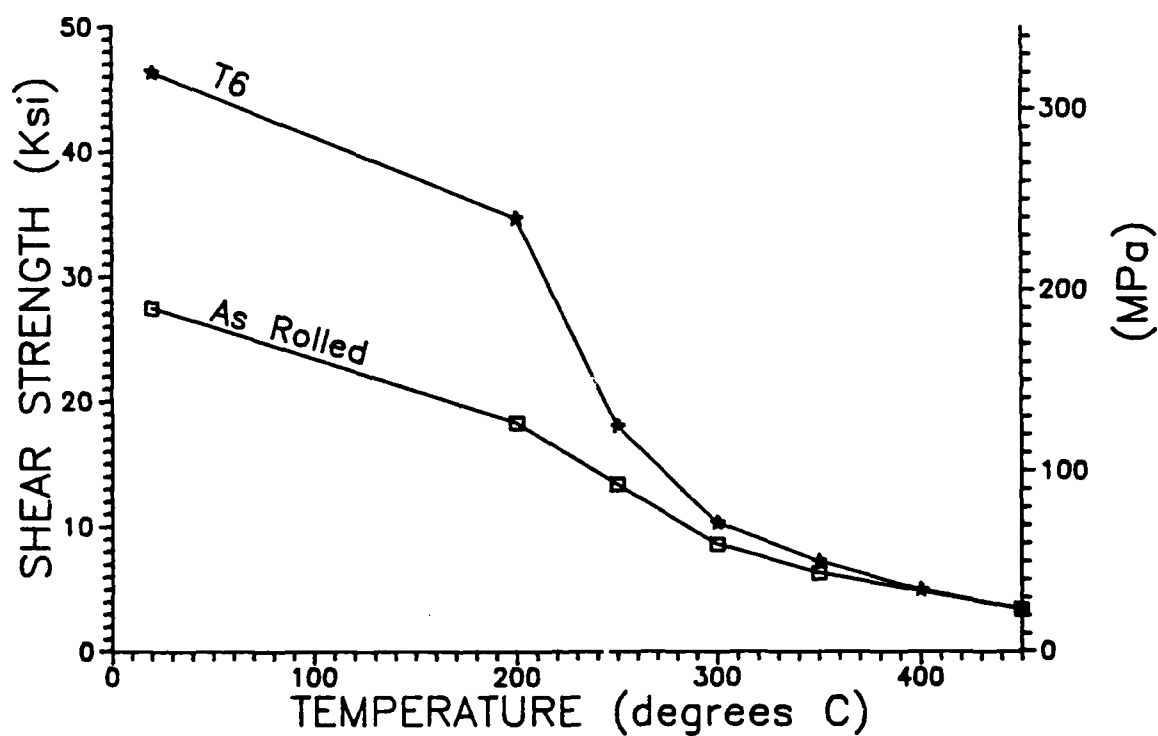
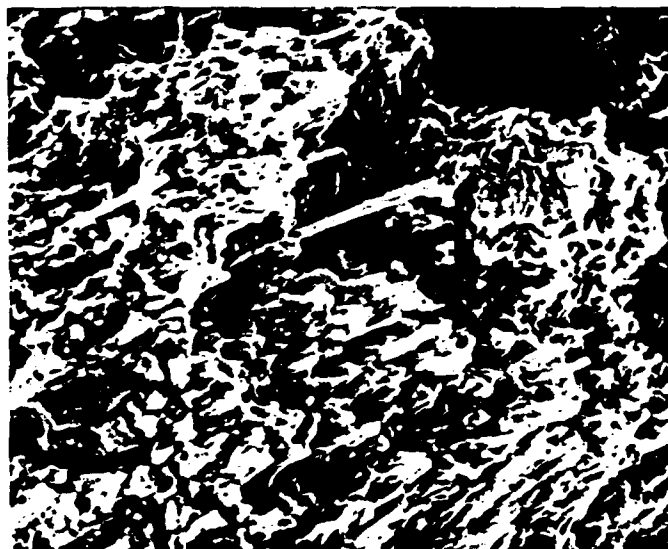


Figure 4. Ultimate shear strength vs. temperature for hot rolled AA2124 15v/o SiCw in the as-rolled and the T6 condition.



5 μ m

Figure 5. SEM of the fracture surface of an AA2124 20v/o SiCw composite tensile tested at 450°C.

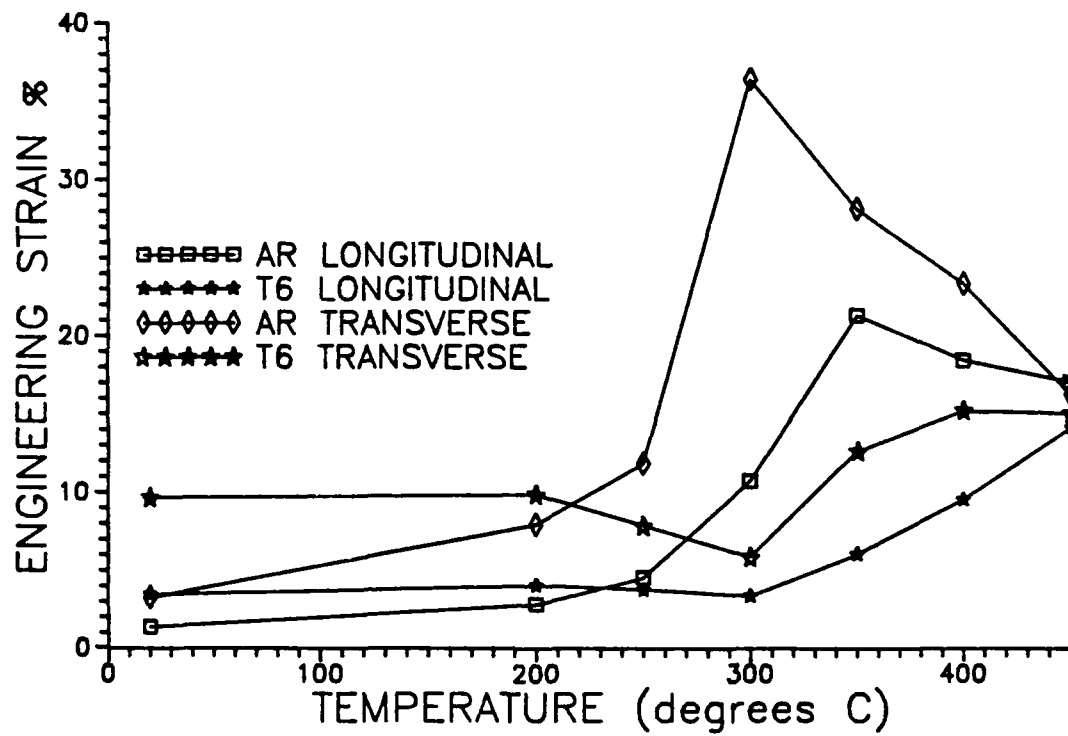


Figure 7. Engineering Strain to failure for hot rolled AA2124 15v/o SiCw in the as-rolled and the T6 condition.

SUBMITTED TO JOURNAL OF MATERIALS SCIENCE

EFFECTS OF MICROSTRUCTURE ON OPTIMUM HEAT TREATMENT CONDITIONS IN METAL MATRIX COMPOSITES

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ABSTRACT

It is well known that the microstructure of metal matrix composite materials is significantly different than that of the unreinforced matrix. Therefore, heat treatments which optimized strength values in the matrix no longer do so in the composite. Thus it is beneficial to find these variations in the heat treatment process, and to pinpoint the microstructural mechanisms responsible. It is known that, in addition to a higher dislocation density, the composite also has a very fine grain size and many whisker interfacial nucleation sites. This study has found that the time required for solutionizing appears to be much shorter in the composite primarily due to the very small grain size. It was also observed that artificial aging was not very effective in the composite since the precipitates nucleated and grew on the whisker interfaces. Therefore, only natural aging was necessary to achieve peak tensile and yield strengths.

1. INTRODUCTION

An area of fundamental importance in metal-matrix composite (MMC) research is the study of response of the composite to heat treatment conditions. Many investigations on whisker reinforced MMCs have utilized heat treatment conditions which were devised for unreinforced matrix material [1-4]. However, it is well known that in order to obtain optimum properties from the composite, variations in heat treatment conditions are required due to the microstructural differences between the composite and the unreinforced matrix. For example it had been found by Christman and Suresh [5] that the time for peak artificial aging in their AA2124-SiC whisker composite was smaller than that in the matrix by a factor of 3. Likewise, Nieh and Karlak [6] had shown that reinforcing B_4C particles in AA6061 reduced the peak aging time by a factor of 3. Similar results were also found in a theoretical investigation by Dutta and Bourell [7].

The more rapid artificial aging kinetics in composites had been attributed to a higher diffusion rate resulting from a higher dislocation density. This high dislocation density was, in turn, due to the 5:1 difference in thermal expansion coefficients of Al and SiC, which was shown by Vogelsang, et al. [8]. Christman and Suresh [5] have found dislocation densities as high as $5 \times 10^{14} \text{ m}^{-2}$ in an AA2124 13v\% SiCw composite, as compared to $5 \times 10^{13} \text{ m}^{-2}$ found in unreinforced AA2124.

However, other microstructural differences also exist between the composite and the unreinforced matrix which affect the results of heat treatments. In order to fully utilize the

potential of whisker reinforced composites it is necessary to develop an understanding of the microstructural mechanisms that control the heat treatment response for these systems. Therefore it was the objective of this study to investigate the effects of composite microstructure on the heat treatment conditions in a AA2124/15v/o SiC whisker composite.

2. EXPERIMENTAL PROCEDURE

2.1 Material

A rolled sheet of AA2124 (SXA24E) reinforced with 15 volume percent (v/o) SiC whiskers was received from Advanced Composite Materials Corporation in Greer, SC. This sheet was hot rolled at a temperature of 438°C to a thickness of 1.8 mm (0.071 inches); approximately an 80 percent reduction. The material was solutionized at 495°C between passes to prevent fracturing. The last pass was performed at room temperature.

2.2 Tensile Tests

Tensile bars of length 7.5 cm were cut from the material. The center 5 cm of these bars were rounded on the lathe with 600 grit SiC paper. These samples were tested using an Instron Tensile Testing Machine at an initial strain rate of 0.00067 s⁻¹.

2.3 Microstructural Studies

The cross section of metallographic samples were prepared using conventional polishing methods followed by a short period of ion milling (about 3 hours) at a voltage of 4 kev and a current of 0.5 mA. With this method, a clean new surface is exposed in which whiskers, voids, precipitates, and in some instances even grain boundaries could be seen clearly on the SEM.

3. RESULTS

3.1 Background

Earlier work had shown that hot rolling significantly reduced the tensile strength of the peak aged AA2124 SiCw composite by overaging the matrix [9]. Large Al_2Cu precipitates (3-5 μm in diameter) grew during the brief cooling period after hot rolling. It was also noted that after a T6 heat treatment (1 hour at 500°C, cold water quench, + 8 hours artificial aging at 150°C, a practice suggested by Advanced Composite Materials Corporation) the tensile strength was regained. This recovery in tensile strength was determined to be due to solutionizing of the large precipitates which nucleated premature failure.

3.2 Solutionizing results

Solutionizing experiments were performed to find the minimum time necessary to dissolve the large precipitates. As-rolled material was solutionized at 500°C for different periods of time, room temperature aged, and then tensile tested. The solutionizing time was taken to be the time between placing the samples in the heated air furnace and the time of removal and immediate cold water quenching. Figure 1 shows the graph of tensile and 0.2% yield strength vs. solutionizing time for the rolled AA2124 15v/o SiCw composite. It can be seen that the precipitates were dissolved and the strength regained in less than 1.5 minutes of solutionizing time, as was also confirmed by microscopic examination. This appears to be somewhat faster than in unreinforced aluminum since the Metals Handbook [10] suggested that an AA2024 sheet of thickness 1.8 mm should be solutionized at a temperature of 495°C for a time of 35-45

minutes in an air furnace or for a time of 25-35 minutes in a salt bath to fully solutionize the precipitates.

3.3 Microstructural observations

Figure 2 is an SEM micrograph oriented parallel to the whiskers showing the precipitates, whisker ends, and the grains in this composite. The grain size was measured using the Mean Intercept Length Method [11]. In this composite the mean intercept length was measured to be 0.5 μm . These grain sizes were also confirmed with the TEM.

3.4 Aging of the Composite

Experiments were also carried out to investigate the aging kinetics of the composite. Natural aging and artificial aging were studied. Figure 3 shows 0.2% yield and tensile strength vs. room temperature aging time in the AA2124/SiCw system. This can be compared to Figure 4, a graph of yield and tensile strength vs. artificial aging time in the same material. It was observed that natural aging for over 24 hours gave yield and tensile strength values very similar to those of material which had been artificially aged at 150°C followed by 24 hours of room temperature aging. These results are in contrast to those of unreinforced AA2014, where peak artificial aging results in yield [12] and tensile [13] strengths quite a bit higher than those obtained from natural aging, as can be seen in Table 1.

Although natural aging and artificial aging resulted in similar strength values in the composite, the ductility values were slightly different. Natural aging resulted in an average strain to failure of 2.6% compared to 3.4% from artificial aging.

On the other hand, the ductility of unreinforced matrix material reacted in the opposite manner. The strain to failure of natural aged material was 22% compared to 10% in the artificially aged material [13].

4. DISCUSSION

The solutionizing experiments appeared to show that the solutionizing rate was more rapid in the AA2124 15v/o SiCw composite than in the unreinforced matrix. This effect seemed to be due to the very small grain size observed. It is known that grain sizes in excess of 1 mm are not uncommon in unreinforced aluminum. However, Jarry et al. [14] have found that the introduction of ceramic whiskers or particles induces a very small and stable grain size (about 1 μm), an effect attributed to the mean spacing and anticrystallization power of reinforcements. The fine grain size observed in the composite results in more grain boundary area, thus the amount of material allowed to diffuse rapidly along the grain boundaries would be increased. Hence, the time necessary to solutionize the material would be decreased.

In order to better show the affect of grain size on diffusivity, a more quantitative analysis is performed. It is well known that the total diffusion in a material can be written as [15]

$$D = D(l) + g(d)D(d) + g(gb)D(gb) \quad (1)$$

where $D(l)$ is the diffusion rate in the lattice, $g(d)$ is the fraction of atoms in the dislocation core, $D(d)$ is the diffusion

rate in the core, $g(gb)$ is the fraction of atoms in the grain boundary, and $D(gb)$ is the diffusion rate in the grain boundary.

The values of $g(d)$ and $g(gb)$ obviously depend on the dislocation density and the grain size in the material. As stated earlier the dislocation density in a similar composite was found to be of the order of $5 \times 10^{14} \text{ m}^{-2}$ [5]. This dislocation density gave a value for $g(d)$ of 0.0005, assuming the dislocation core has a cross sectional area of 100 \AA^2 [16]. A model using cubic grains $0.5 \text{ }\mu\text{m}$ on edge with a grain boundary thickness of 3 atoms [17], gave a value for $g(gb)$ of 0.007.

The diffusion constants were found to be the following:

lattice diffusion [18],

$$Q = 32.3 \text{ Kcal/mol}$$

$$D_0 = .647 \text{ cm}^2/\text{sec}$$

dislocation diffusion [19],

$$Q = 19.6 \text{ Kcal/mol}$$

$$D_0 = .028 \text{ cm}^2/\text{sec}$$

grain boundary diffusion (estimate) [16]

$$Q \approx 16 \text{ Kcal/mole}$$

$$D_0 \approx .03 \text{ cm}^2/\text{sec}$$

Figure 5 shows the Diffusivity vs. Temperature results for these values. It is apparent that the very small grain size in the composite leads to grain boundary controlled diffusion even at high temperatures.

With an estimate of the spacing of the precipitates, calculations could be made in order to get an approximation of the time it should take the precipitates to be dissolved in the material. The precipitates in the overaged material could be seen using backscattered electron imaging in the compositional mode. Figure 6 shows these precipitates in as-rolled composite

material. The precipitates are approximately 3-5 μm in diameter with a spacing of about 10 μm . Therefore, the precipitates should be dispersed in roughly the amount of time it takes for copper to diffuse a mean distance of about 5 μm at the solutionizing temperature. This distance can be estimated with the equation [20]

$$d \approx \sqrt{Dt} \quad (2)$$

where d is the mean diffusion distance, D is the diffusivity, and t is the time allowed for diffusion. Therefore, with grain boundary controlled diffusion it should theoretically take only about 1 minute to dissolve the precipitates at 500°C, which agrees well with the earlier observations. With self diffusion it would theoretically take about 7.5 minutes, although this could not be checked since unreinforced SXA24E alloy was not available. It should also be noticed that since the grain size (0.5 μm) was much smaller than the precipitate size (3-5 μm), many grain boundaries intersected the large precipitates (as can be seen in figure 2). Thus, grain boundary diffusion would indeed have a significant effect on the solutionizing rate of the precipitates.

The aging experiments (Table 1) showed that peak natural aging and peak artificial aging gave virtually identical tensile and yield strength values in the composite material. Whereas in unreinforced aluminum alloys, peak artificial aging was found to give much higher strength values than natural aging [12,13]. Thus artificial aging did not appear to be as effective in the

composite material. This effect appeared to be a result of precipitate nucleation on the whiskers. In unreinforced aluminum-copper alloys, aging at 150°C results in the growth of θ'' precipitates, which are similar in shape to GP zones only larger [21]. GP zones are described as disk-like copper rich regions about 9 nm in diameter that homogeneously nucleate and grow parallel to the {100} planes [22]. However, in the composite material aged at 150°C, precipitates appeared to heterogeneously nucleate and grow at the whisker interfaces, as can be seen in Figure 7. This interfacial nucleation may have had an adverse effect on strength by embrittling the interfacial region. In addition, not as many θ'' precipitates would be allowed to form in the matrix, resulting in reduced precipitate strengthening. This reduced precipitate strengthening in the T6 material may have also been responsible for the slightly higher ductility found in this material, as compared to the naturally aged material.

Although others have found that artificial aging was more rapid in composites than in unreinforced material [5-7], this does not imply that room temperature aging would be faster. In fact, it can be seen in Figure 8 that the rate of room temperature strengthening was nearly identical for the composite and unreinforced AA2024 [23]. Aging done at temperatures near or below 100°C strengthens the alloy through the formation of GP zones [24]. Since these GP zones nucleate homogeneously and are very small and close together, their rate of growth will not depend on the grain size or the dislocation density of the

composite. Thus, one would expect the rate of room temperature strengthening to be very similar in both the reinforced and unreinforced aluminum alloy, as was shown.

5. CONCLUSIONS

Heat treatment conditions appear to be different in metal matrix composites than in the matrix alloys alone. This study has found that the time necessary for solutionizing appeared to be much shorter in the composite primarily due to a very small grain size. The precipitates were completely dissolved in less than 1.5 minutes at 500°C. Therefore, the normally applied T6 solutionizing time of 1 hour is not necessary and much time and energy can be saved.

It was also observed that artificial aging was not very effective in the AA2124 15v/o SiCw composite since the precipitates nucleated and grew on the whisker interfaces. Therefore, only natural aging was necessary to get peak tensile and yield strength values, although a slight decrease in ductility was noticed. The rate of room temperature aging, however, was observed to be nearly identical in both the composite and the unreinforced alloy.

Acknowledgements

The authors appreciate the support of the Office of Naval Research, Dr. Steven G. Fishman, Scientific Officer, under grant #N00014-85-K0179.

REFERENCES

1. S.V. Nair, J.K. Tien, R.O. Bates, *Int. Met. Rev.*, 30 (1985) 275.
2. T.G. Nieh and R.F. Karlak, *J. of Mat. Sci. Letters*, 2 (1983) 119.
3. D.L. McDanel, *Met. Trans.* 16A, (1985) 1105.
4. Y. Fujita, H. Fukumoto, Y. Kurita, *ICCM & ECCM*, 2 (1987) 2.340.
5. T. Christman, S. Suresh, Brown University Report #NSF-ENG-8451092, 1987.
6. T. G. Nieh and R. F. Karlak, *Scripta Met.*, 18 (1984) 27.
7. I. Dutta, D. Bourell, D. Latimer, *J. of Comp. Mat.*, 22 (1988) 829.
8. M. Vogelsang, R. J. Arsenault and R. Fisher, *Met. Trans.*, 17A (1986) 379.
9. R. Schueller and F. Wawner, "The Effects of Hot Rolling on AA2124 15% SiCw Composites," To be published in *Journal of Comp. Mat.*, Oct, (1990).
10. Metals Handbook, ASM, Metals Park, Ohio, 9th Ed., 4 (1985) 684.
11. Metals Handbook, ASM, Metals Park, Ohio, 8th Ed., 8 (1973) 45.
12. Aluminum: Properties and Physical Metallurgy, Ed. J. E. Hatch, American Society for Metals, Metals Park, Ohio, p. 138, (1984).
13. Aluminum: Properties and Physical Metallurgy, Ed. J. E. Hatch, American Society for Metals, Metals Park, Ohio, p. 362, (1984).
14. Ph. Jarry, W. Loue, J. Bouvaist, *ICCM & ECCM*, 6th International conference on composite materials, 2 (1987) 2.359.
15. P. G. Shewmon Diffusion in Solids, McGraw Hill, NewYork, (1963) 176.
16. A.L. Ruoff, Material Science, Prentice Hall, Inc., New Jersey, (1973) 465.
17. M.A. Meyers and K.K. Chawla, Mechanical Metallurgy, Prentice Hall Inc., Englewood Cliffs, New Jersey, p. 276, 1984.
18. N.L. Peterson and S.J. Rothman, *Physics Rev.*, B1(8) (1970) 3264.
19. N. A. Gjostein, Diffusion, ASM, Metals Park, Ohio, (1973) 263.
20. A.L. Ruoff, Material Science, Prentice Hall, Inc., New Jersey, (1973) 457.
21. A. Guinier, *Acta Cryst.*, 5, (1952) 51.
22. R.B. Nicholson and J. Nutting, *Phil. Mag.*, 3, (1958) 531.
23. Metals Handbook, ASM, Metals Park, Ohio, 9th Ed., 4 (1985) 698.
24. G.W. Lorimer, Precipitation Processes in Solids, ed. K.C. Russel and H.I. Aaronson, The Met. Society of AIME, (1978) p.87-119.

TABLE 1

Peak Yield and Tensile Strengths for Unreinforced AA2014 and AA2124 15% SiCw Composites After Natural Aging and Artificial Aging.

| MATERIAL | AGING CONDITION | PEAK 0.2% YIELD STRENGTH | | PEAK TENSILE STRENGTH | |
|-----------------|--------------------|-----------------------------|-------|--------------------------|-------|
| | | (Ksi) | (MPa) | (Ksi) | (MPa) |
| AA2014 | NATURAL AGED | 42 | 290 | 60.9 | 420 |
| AA2014 | AGED AT 150°C | 63 | 435 | 68.2 | 470 |
| AA2124 15% SiCw | NATURAL AGED | 66.2 | 456 | 106 | 733 |
| AA2124 15% SiCw | AGED AT 150°C | 72.8 | 502 | 105 | 724 |

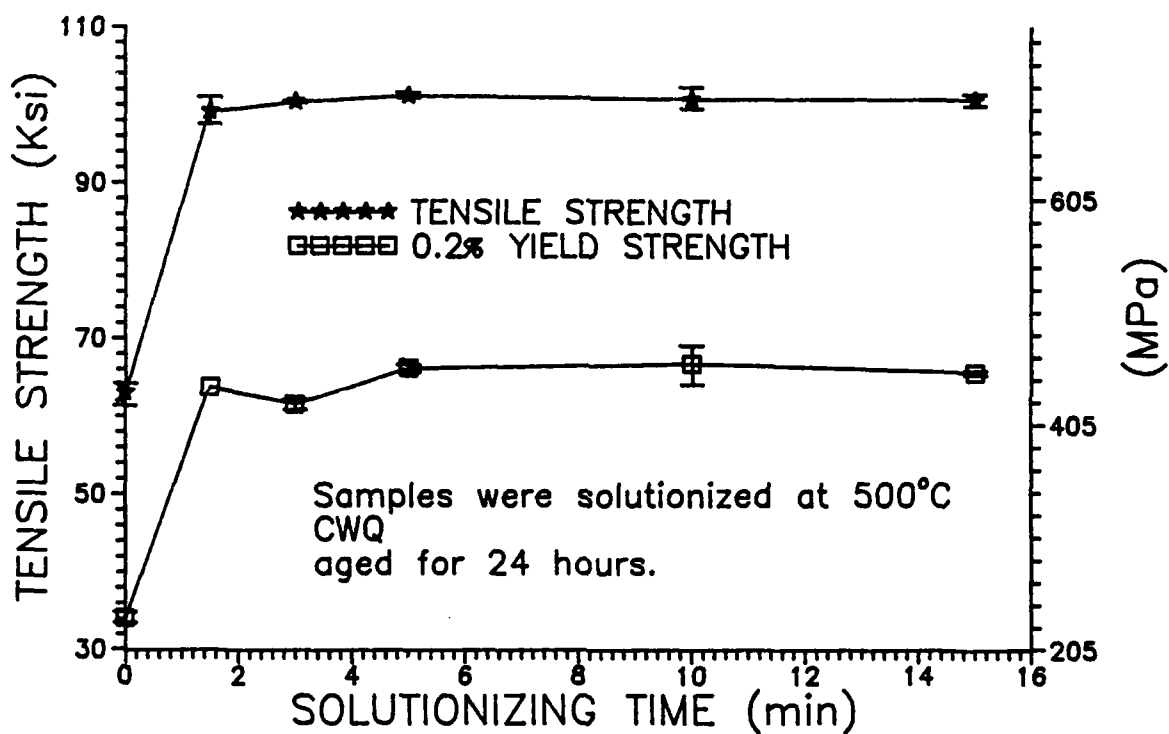


Figure 1. Tensile and 0.2% Yield strength vs. Solutionizing time for hot rolled AA2124 15v/o SiCw composite.

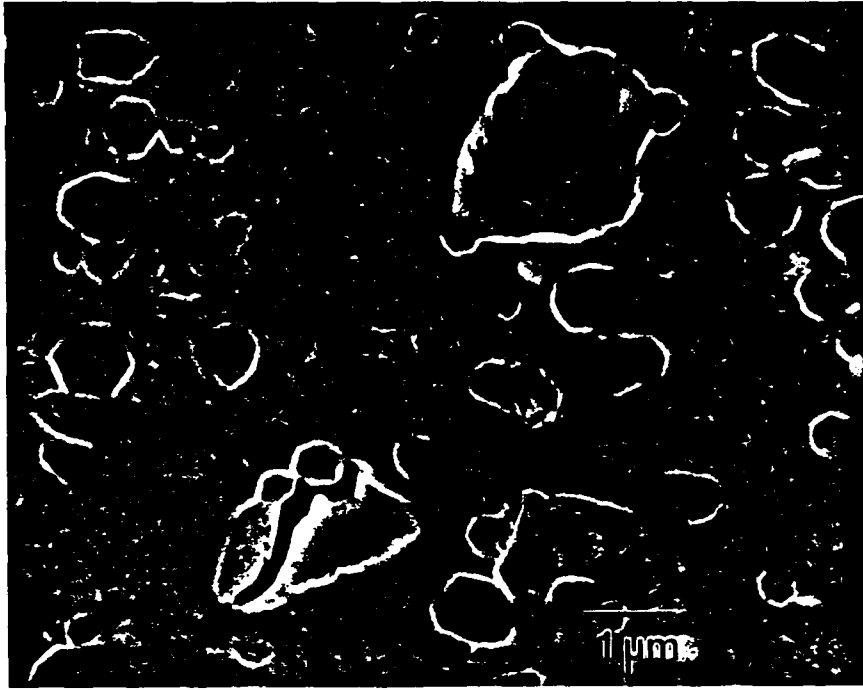


Figure 2. SEM micrograph parallel to whiskers, showing the grain size, the whisker ends and the precipitates.

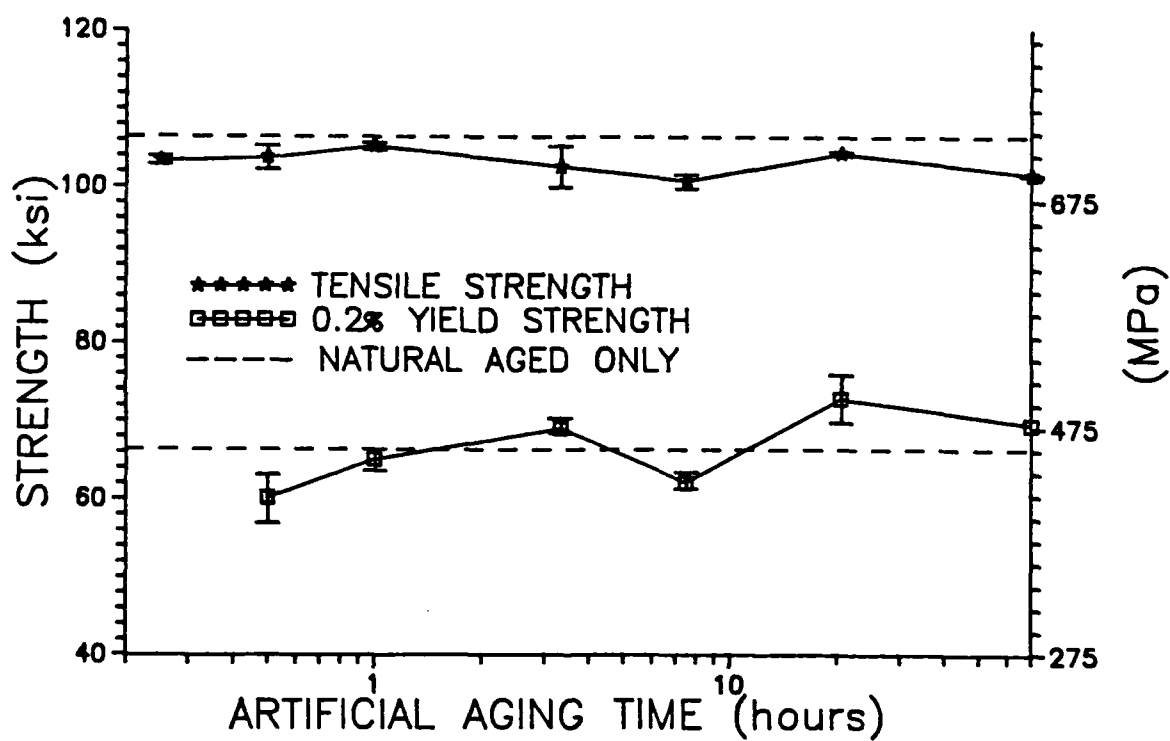


Figure 4. Tensile and 0.2% yield strength vs. aging time at 150°C. Artificial aging was followed by 24 hours of room temperature aging.

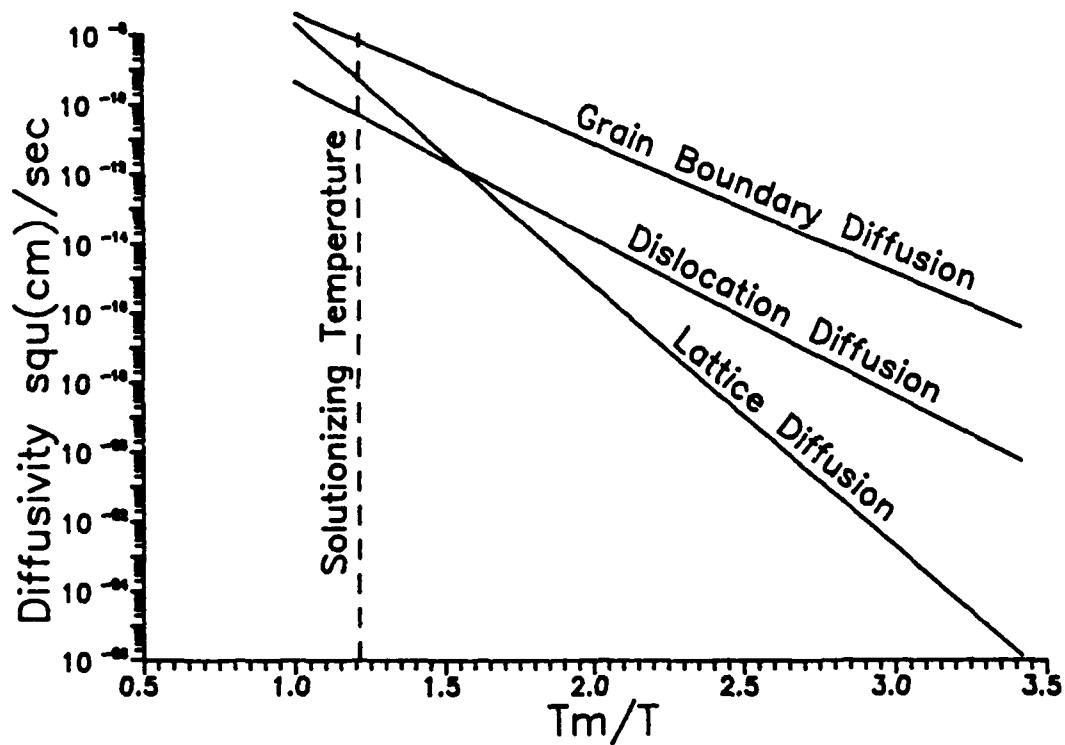


Figure 5. Diffusivity of copper in aluminum at various temperatures, assuming a grain size of 0.5 μm and a dislocation density of $5 \times 10^{14} \text{ m}^{-2}$.

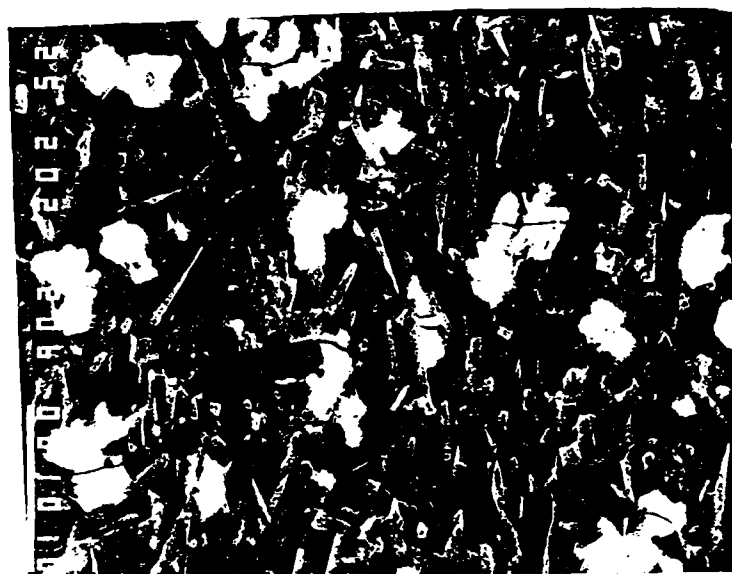


Figure 6. SEM backscattered micrograph showing the size and spacing of the large precipitates.



0.2 μm

(a)



0.3 μm

(b)

Figure 7. Small spherical precipitates at the whisker interfaces in the material aged at 150°C for 8 hours.

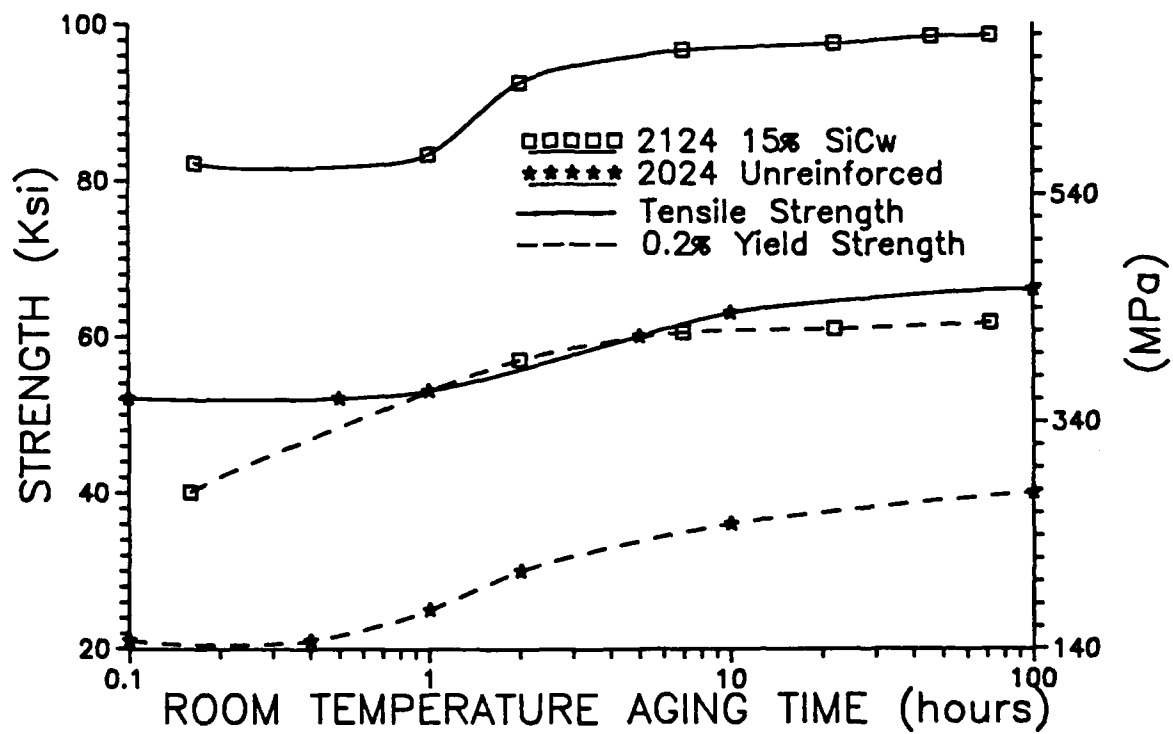


Figure 8. Rate of room temperature strngthening in the reinforced and the unreinforced matrix material.

ADDITIONAL PUBLICATIONS/PRESENTATIONS

- Harris, C.R., and Wawner, F. E., "Influence of Thermomechanical Deformation and Transverse Fracture in SiC_w/Al Composites," 9th Annual Discontinuous Reinforced Metals Working Group (Park City, UT, January 6-8, 1987); Conference Proceedings Series MMCIAC No. 000695, Dec 1988, pp. 527-545.
- Schueller, R., and Wawner, F., "An Analysis of High Temperature Behavior of Discontinuously Reinforced Al Alloys," 10th Annual Discontinuous Reinforced Metals Working Group Meeting (Park City, UT, January 4-8, 1988); Conference Proceedings Series MMCIAC No. 000696, Jan 1989, pp. 129-139.
- Wawner, F. , "Boron and Silicon Carbide/Carbon Fibers," Chapter 8, Fibre Reinforcements for Composite Materials, A. R. Bunsel, editor (Elsevier, Amsterdam, 1988), pp. 371-426.
- Schueller, R., and Wawner, F. E., "An Analysis of the Effects of Hot Rolling Discontinuously Reinforced Al Alloys," 66th Annual Meeting of Virginia Academy of Science (University of Virginia, Charlottesville, May 25-27, 1988).
- Schueller, R. and Wawner, F., "A Study of the Effects of Hot Rolling on AA2124/ SiC Whisker Composites," 11th Annual Discontinuously Reinforced MMC Working Group Meeting, Feb. 14-16, 1989, Park City, UT; Conference Proceedings Series MMCIAC No. 716, Jul 1989, pp. 151-176.
- Schueller, R. and Wawner, F., "The Effects of Hot Rolling on AA2124/ SiC . Whisker Composites," presented at 67th Annual Meeting of Virginia Academy of Science (Virginia Commonwealth University, Richmond, VA, May 24-26, 1989).
- Schueller, R. and Wawner, F., "A Study of the Effects of Hot Rolling and Heat Treatments on Discontinuously Reinforced Al-Alloys," presented at Materials Week '89, ASM International, October 2-5, 1989, Indianapolis, IN.
- Wawner, F., "Discontinuously Reinforced Metal Matrix Composites: Microstructure and Mechanical Property Correlations," Encyclopedia of Composites, S. Lee, Editor, VCH Publishers, New York, in press.

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