The Effect of Cryogenic Cooling on the Tensile Properties of Metal-Matrix Composites

JOHN GAYDA AND L. J. EBERT

Tensile specimens machined from metal-matrix, oriented-fiber composites (aluminum alloy reinforced with high strength stainless steel wire) were heated to 260°C and cooled in air to produce a tensile residual stress state in the matrix. Some of the test pieces were cooled to the temperature of boiling nitrogen, held at temperature for fifteen minutes, and then air warmed to room temperature. All test pieces were subsequently strain cycled in tension and the resulting stress-strain behavior was recorded. The results indicated cryogenic refrigeration extended the first stage (totally elastic) behavior of these materials. It was shown that the beneficial effects of the cryogenic treatment resulted from an alteration of the residual stress state brought about by plastic flow of the matrix. Finally, it was shown that these effects could be computed by rigorous analytical methods.

THE high cost of oriented fiber composite materials, together with the need for high performance which justifies their use, make it mandatory that these materials come as close to developing their full potential in mechanical properties as is possible. The elastic modulus and elastic limit (or yield strength) are important mechanical properties to those who must design with them. Thus, the attainment of high values for both of these properties takes on critical importance.

The intrinsic nature of the components of the composite (high strength, high modulus fibers, and lower strength, lower modulus matrices) dictate a three stage response of these materials to mechanical loading—totally elastic, elastic fibers with plastic matrix, and totally plastic (if the fibers are capable of plastic flow). The elastic limit is determined by the onset of plastic flow in the matrix component, while the yield strength is taken as the stress which produces some predetermined minimal amount of plastic flow in the matrix.

Unfortunately, it is almost never possible to achieve the maximum elastic response of which composites are capable because of the presence of residual stresses which result from fabrication. These stresses may be sufficiently high that the totally elastic response is never observed in some composites. The situation is further complicated by the fact that the standard stress relieving operations which are effective in homogeneous materials are almost completely ineffective on composite materials. Hence, the presence of residual stresses constitutes a severe limitation to the use of these materials.

While the residual stresses can never be removed from composite materials, it has been amply demonstrated that their relative intensities can be altered in given directions by the addition of small amounts of prestrain.[1-3] As a matter of fact, it is possible to alter the relative intensities to the degree that the deviatoric component of the residual stress state become compressive in the direction which will be subjected to tension in use. Hence, the residual stresses, as altered by prestrain, can actually be beneficial, rather than deleterious. Further, it has been shown that the beneficial effect of prestrain can be predicted by rigorous analytical methods.

While mechanical prestrain has been shown to be quite effective in reducing or removing the adverse effect of residual stresses, it is not always possible or practical to prestrain composite hardware. Therefore, other measures must be found if composite materials are to achieve their full potential as high performance materials.

In seeking other remedial measures, the significant differences in thermal expansion values between fiber and matrix components suggested the use of temperature increments or decrements as a method of introducing plastic strain into the matrix. The use of temperature decrements (cryogenic cooling) seemed more promising since tensile residual stresses are generated on cool down from elevated temperature.

The results of a study on the use of cryogenic cooling to reduce the adverse effects of residual stresses in oriented fiber composites are reported herein. The study included both experimental and analytical approaches.

MODELING COMPOSITE BEHAVIOR

In order to develop remedial measures for controlling residual stresses, it is necessary to know the intensity and sign of the residual stresses in all three principal directions. Further, it is necessary to know the distribution of the residual stress state from fiber-to-fiber through the matrix. Since all viable experimental methods for residual stress determinations are limited to surface measurements, or are completely destructive, analytical means for determining residual stress data must be used.

Two systems have been developed previously for this purpose. The first, based on finite element techniques,[4] is most effective for thin sheet. The second, which is known as the concentric cylinder model,[5-7] is most effective for plate and bar materials.

JOHN GAYDA and L. J. EBERT are Graduate Student and Professor of Metallurgy, respectively, Case Western Reserve University, Cleveland, OH 44106.

Manuscript submitted August 9, 1978.

ISSN 0360-2133/79/0312-0349$00.75/0

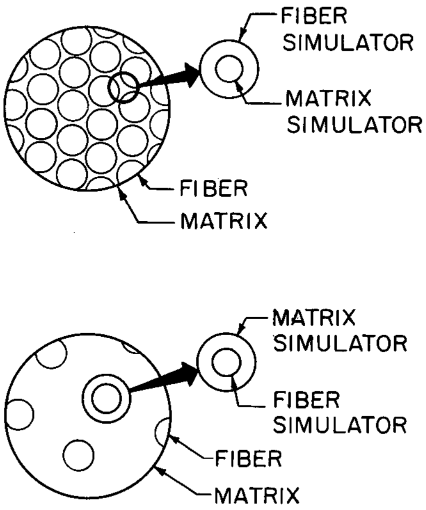

Fig. 1—Conceptual development of the concentric cylinder model for close-packed (top) and loosely-packed (bottom) composites.

These analytical methods developed by Ebert and colleagues have been used successfully under a variety of conditions. The techniques require a knowledge of the component stress-strain curves, volume fraction, thermal expansion coefficients, and prior residual stress state.

Since the primary interest of this study related to plate material, the concentric cylinder model was employed. This model is based on the following concept. The mechanical behavior of a composite can be modeled analytically by using the principles of applied mechanics, if the composite structure is simulated by a two-piece concentric cylinder, in which one member is the fiber simulator and the other member is a matrix simulator. Figure 1 shows how close and loosely packed composites are modeled using this concept. To simulate composite behavior, one solves the corresponding problem which describes the behavior of the simple concentric cylinder. A more detailed description of the concentric cylinder model can be found in several papers by Ebert, Hamilton, and Hecker.[6,7]

EXPERIMENTAL PROCEDURE

6061 aluminum alloy–stainless steel, unidirectional composite plates, approximately 1.27 cm thick with 60 and 34 vol pct (fiber content by volume) of 0.33 mm stainless steel wire were fabricated by DWA Incorporated for this study. The plates were manufactured by alternately winding heavily cold worked stainless steel wire and aluminum foil on a large mandrel to create a ''wide tape'' preform. This preform was cut into the appropriate size sheets, which were laid up to give the desired thickness. The pack was then hot-pressed under vacuum at 520°C for thirty min using a pressure of 28 MPa. Buttonhead tensile specimens, shown in Fig. 2, were subsequently machined from these plates.

To produce a known rational residual stress state, the tensile specimens were heated to 260°C, held at this temperature for thirty min, and then air cooled. The purpose of this treatment was twofold. First, the anneal at 260°C was designed to eliminate any prior residual stresses by creep deformation of the aluminum. The time and temperature were held to a minimum to prevent reactions at the fiber-matrix interface which might weaken the composite. Second, the air cool generates a tensile residual stress state in the matrix which can be computed.

Half of the annealed specimens were subsequently refrigerated at $-196°C$ by immersion in liquid nitrogen. After fifteen min, the specimens were removed and allowed to warm in still air. The refrigeration was expected to produce a compressive residual stress state in the matrix, thereby increasing the primary yield point of the composite.

All specimens were subsequently strain cycled in tension. The testing was done with an Instron tensile machine (Model TT-C) at a crosshead speed of 0.508 mm per min. Axial strain measurements were made with electrical resistance strain gages.

COMPOSITE BEHAVIOR

The axial stress-strain curves for all conditions and volume fractions studied are presented in Figs. 3 through 8. In each instance, the solid line represents experimental data and the circles represent predicted behavior based on the concentric cylinder model.

Examination of the data reveals the following. The 60 vol pct specimens exhibit Stage I (elastic fiber-elastic matrix) and Stage II (elastic fiber-plastic matrix) behavior; however, the primary yield point of the refrigerated specimens is five times that of the annealed material. Consistent with the above, one sees the amount of permanent strain associated with the refrigerated material is less. The response on reloading is essentially linear. Note the lack of a pronounced hysteresis loop. This indicates the matrix behaves elastically upon reloading (and unloading). While work hardening of the matrix does occur, it is not the primary strengthening mechanism. The increase of the primary yield point, be it a result of refrigeration or mechanical prestraining, is a direct

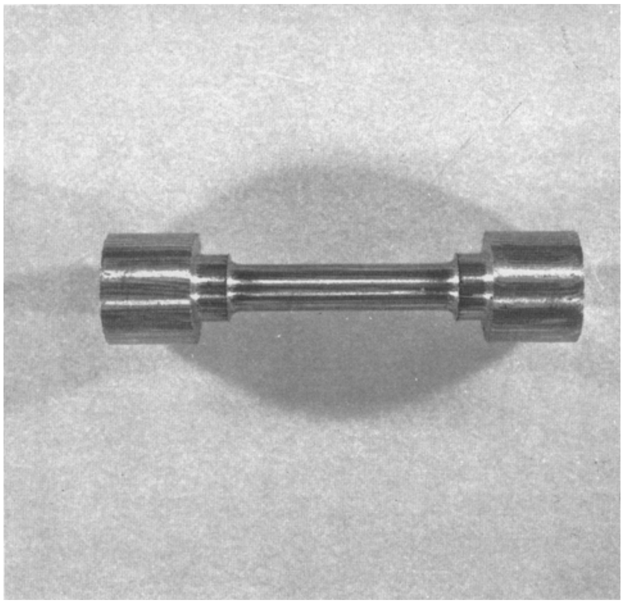

Fig. 2—Machined tensile specimen used in this investigation. Magnification 1.3 times.

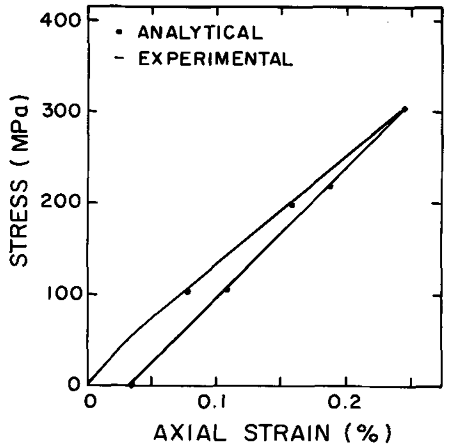

Fig. 3—Stress-strain curve of the annealed material. 60 vol pct.

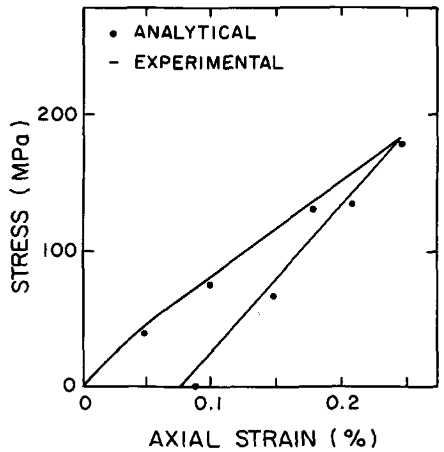

Fig. 6—Stress-strain curve of the annealed material. 34 vol pct.

consequence of a compressive residual stress state created in the matrix. This concept will be explained in more detail later.

The 34 vol pct material was also strengthened by prestraining and refrigeration; however, composite response was different from the 60 vol pct material in several respects. First, on reloading a substantial hysteresis loop is observed. Second, the prestrained material exhibits a yield point on unloading (see Fig. 7). Both phenomenon are a direct result of the decreased fiber content. As before, the increase in composite strength on refrigerating or prestraining, is primarily due to a compressive residual stress state in the matrix; however, work hardening of fiber and matrix makes a substantial contribution to composite strength at the lower fiber fraction.

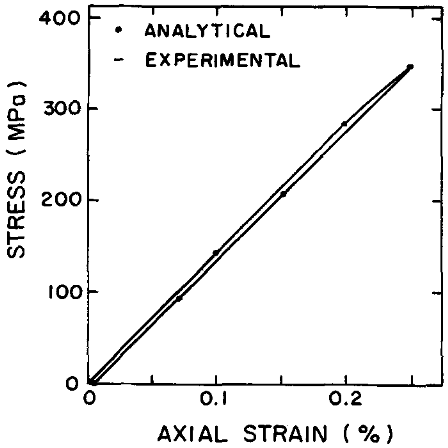

Fig. 4—Stress-strain curve of the prestrained material. 60 vol pct.

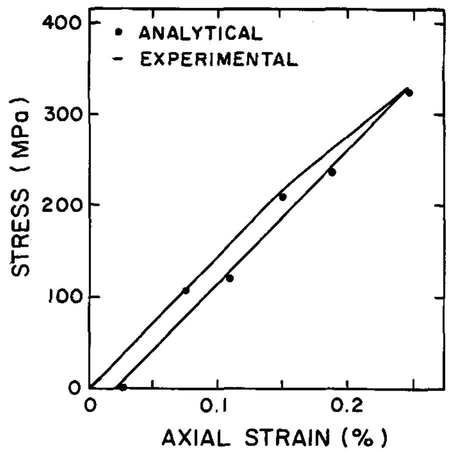

Fig. 5—Stress-strain curve of the refrigerated material. 60 vol pct.

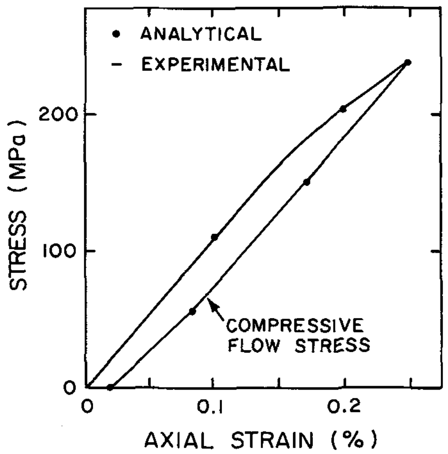

Fig. 7—Stress-strain curve of the prestrained material. 34 vol pct.

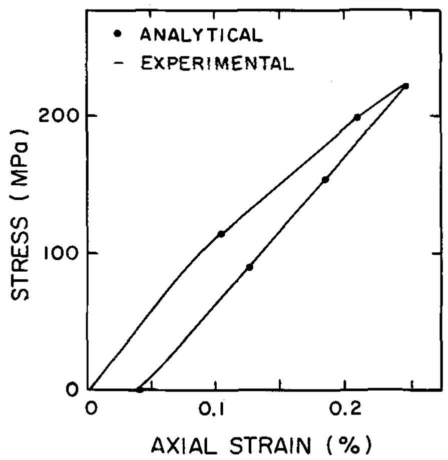

Fig. 8—Stress-strain curve of the refrigerated material. 34 vol pct.

DEVIATORIC COMPONENTS OF THE RESIDUAL STRESS STATE

The full importance of the prior residual stress distribution and its affect on tensile behavior can best be seen by examining the deviatoric component of the stress in the fiber direction, the direction in which the composite will be loaded in service. The deviators are computed by subtracting the hydrostatic component (average of the principal stresses) from the individual stresses in all three principal directions. It should be noted that the transverse components of the residual stress distribution were not negligible, and at times actually exceed the magnitude of the axial component. A typical residual stress distribution from which the deviatoric stresses are calculated is shown in Fig. 9. The axial deviator of the matrix is of special interest, because it is the low flow stress of this component which will limit the elastic behavior of the composite.

The deviators associated with the residual stress distribution in the fiber direction were plotted for all conditions. The results are presented in Figs. 10 and 11. Examining these distributions, certain characteristics can be associated with each condition whether one considers the high or low fiber fraction. First, the annealed condition is characterized by a tensile stress deviator in the matrix and a compressive stress deviator in the fiber. Second, refrigeration is found to diminish the magnitude of the original distribution. Third, prestraining produces a condition in which the matrix is characterized by a compressive stress deviator while the fiber stress becomes tensile. The effect of prestraining and refrigeration is similar although the magnitude is decisively different.

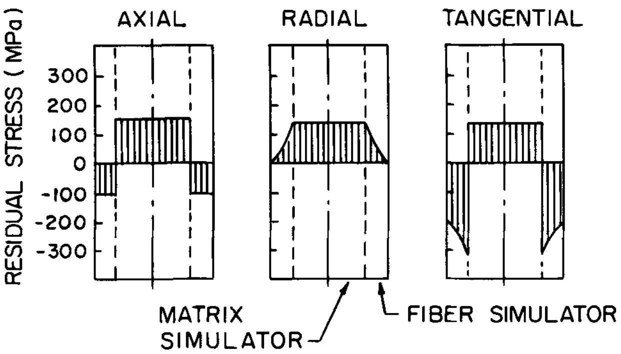

Fig. 9—A typical residual stress distribution which was used to calculate the deviatoric stress components in the fiber direction.

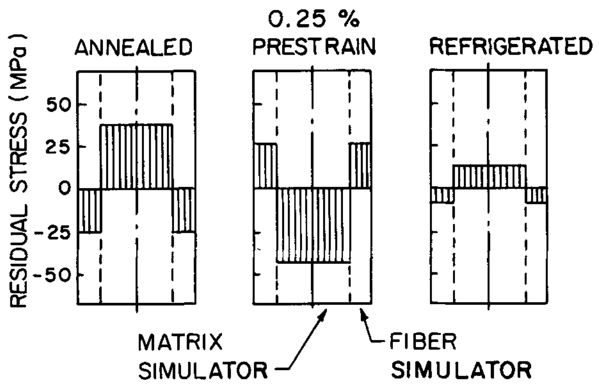

Fig. 10—The deviatoric stress components in the fiber direction associated with the three material conditions studied herein. 60 vol pct.

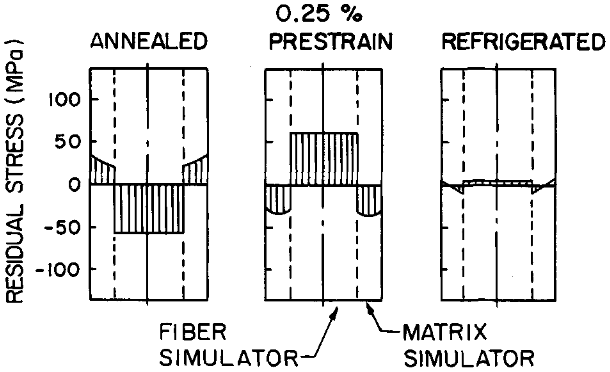

Fig. 11—The deviatoric stress components in the fiber direction associated with the three material conditions studied herein. 34 vol pct.

MECHANISMS

Tensile behavior of metal-matrix composites is most easily visualized by using the concepts discussed in the previous section. Three cases will be considered in the following paragraphs. They are a) cooling from elevated temperatures, b) refrigeration to cryogenic temperatures and c) prior tensile prestraining.

Cooling from elevated temperatures will produce a tensile deviator in the matrix if its coefficient of thermal expansion exceeds that of the fiber. Since the two components are bonded together, the fibers restrict the free *contraction* of the matrix causing a tensile stress deviator therein. To maintain a balance of forces, a compressive stress deviator is generated in the fiber for the same reason. Analytically, the materials studied gave rise to situations in which the matrix flowed on cooling to room temperature. On loading the composite, Stage I behavior is substantially reduced or eliminated since the matrix is at or near the tensile yield point.

Refrigeration to cryogenic temperatures further increases the absolute magnitude of the matrix deviator, since the composite temperature continues to decrease. However, the matrix deviator becomes less tensile when the composite is removed from the cryogenic environment. This occurs because the matrix *expansion* is now restrained by the fibers which have a lower coefficient of thermal expansion. The fiber deviator must become less compressive to maintain a balance of forces. If the matrix does not flow on cooling, the refrigeration will not change the residual stress distribution and subsequent tensile behavior since the situation would be fully reversible, *i.e.* the change in deviatoric stress on heating and cooling is opposite and equal. When the matrix does flow on cooling and behaves elastically on warming, the moduli are different and, therefore, the process is not reversible, *i.e.* the change in deviatoric stress on heating and cooling is opposite but unequal. Subsequent tensile behavior of the composite may, therefore, do one of two things on refrigerating. If flow has occurred, the primary yield point will increase since the matrix deviator is now less tensile. If the matrix did not flow, the refrigeration would not change the primary yield point since the matrix deviator remains unchanged. In this investigation, the former situation was observed experimentally and analytically.

Prestraining tends to increase the primary yield point of metal-matrix composites by producing a residual stress state in which the matrix deviator is compressive. This condition is produced by prestraining the composite beyond the primary yield point. On unloading, the matrix behaves elastically, and therefore, as was the case with refrigeration, the situation is not reversible, resulting in a matrix deviator which is less tensile. If the composite is prestrained sufficiently, the matrix deviator may reach the compressive flow strength (of the matrix) on unloading. This condition places an upper limit on the primary yield point in the absence of any strain hardening which is approximately equal to twice the matrix flow stress multiplied by the ratio of the composite and matrix moduli, E_c/E_m. Consequently, increasing the initial load beyond this critical level does little to raise the primary yield point.

CONCLUSIONS

On the basis of the data presented herein, it seems quite clear that the analytical model developed by Ebert and colleagues is capable of predicting the mechanical behavior of oriented fiber, metal-matrix composites under a variety of conditions. Further, the work has shown that refrigeration to cryogenic temperatures is instrumental in extending the stress range of totally elastic behavior for these materials. The mechanism which produces this affect can be understood by examining the change in the existing residual stress state.

ACKNOWLEDGMENTS

The authors are grateful for the support of the National Science Foundation, which made the study described herein possible. Specifically, the cooperation and assistance of Dr. Roman J. Wasilewski is gratefully acknowledged.

REFERENCES

1. R. J. Fedor and L. J. Ebert: *J. Eng. Mater. Technol.*, 1973, vol. 95, p. 69.
2. R. W. Heckel, J. J. Zaehring, and H. P. Cheskis: *Met. Trans.*, 1972, vol. 3, p. 2507.
3. M. Kh. Shorshorov, L. M. Ustinov, Yu. G. Kuznetsov, L. V. Vinogradov, and V. I. Jamnova: *Composities*, 1976, vol. 7, p. 17.
4. P. K. Wright and L. J. Ebert: *Met. Trans.*, 1972, vol. 3, p. 1645.
5. L. J. Ebert, S. S. Hecker, and C. H. Hamilton: *J. Compos. Mater.*, 1968, vol. 2, p. 458.
6. S. S. Hecker, C. H. Hamilton, and L. J. Ebert: *Scr. Met.*, 1969, vol. 3, p. 793.
7. S. S. Hecker, C. H. Hamilton, and L. J. Ebert: *J. Mater.*, 1970, vol. 5, p. 868.