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The Effect of Temperature on the Impact
Behavior of TiB₂ Reinforced XDTM-TiAl
Intermetallic Matrix Composites

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A study was performed to determine the effect of temperature on the impact behavior of two XD TM processed TiB ₂ reinforced TiAl intermetallic composites (IMC's). An existing instrumented tup drop tower apparatus was modified for the program. The IMC compositions were Ti-49 at.% Al + 5 vol.% TiB ₂		

and Ti-48 at % Al + 10 vol.% TiB₂. The specimens were notched to an a/w ratio of 0.2. Details and test procedures for the modified apparatus were presented. Test temperatures ranged from -192°C to 1100°C. Impact toughness and maximum bending stress for both IMC's decreased from room temperature to temperatures as high as 1100°C. An important experimental observation which supports these results is the change from transgranular cleavage to intergranular fracture at the 760°C test temperature. Also noted is the possible contribution to decreasing toughness of unfavorable difference in coefficient of thermal expansion between TiB₂ and the matrix with increasing temperature. Data repeatability and verification of the ability to observe high temperature brittle-to-ductile transition behavior established confidence in the modification of the existing instrumented tup apparatus.

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INTRODUCTION

High performance propulsion systems and structures for hypersonic vehicles require advanced materials. These materials must have strength, ductility, toughness and resistance to oxidation at high temperatures. One family of high temperature materials are intermetallic compounds. An early summary and review of their properties was provided by Westbrook (1). He noted their principal mechanical characteristics were brittleness and extreme hardness. Also, he pointed out the very early work of Tamman and Dahl (2) in which they performed impact tests to classify intermetallic compounds on the basis of the temperature dependence of their ductile-brittle behavior. They observed low values of room temperature ductility and impact toughness.

More recently, for the high strength-to-weight ratio and stiffness required for aerospace applications, titanium aluminides, such as, Ti_3Al and $TiAl$ were identified as having the development potential to meet high temperature requirements. These aluminides are designated α_2 and γ , respectively. Early work on the elevated temperature deformation and fracture of γ -aluminides was reported by Lipsitt et al (3,4). They indicated a ductile-to-brittle transition temperature (DBTT) of about $700^\circ C$. The γ -aluminides, however, have more sensitivity to room temperature brittleness than the α_2 alloy. Activity on the α_2 - Ti_3Al type alloys followed and the most recent effort is summarized in the work of Blackburn and Smith (5). More recent activity on titanium aluminides has been summarized by Kim (6). He reports on all their attributes, but still notes their low fracture toughness at low to intermediate temperatures. He notes that activity in the last several years has provided some success through chemistry modification and microstructure control by improved powder metallurgy processing.

During the time that intermetallics were under development, success in metal matrix composites (i.e., metals reinforced with ceramic particles) was accomplished.

The development of an intermetallic matrix composite (IMC), therefore, is an obvious extension. A technology to achieve IMC's was recently accomplished by the Martin Marietta proprietary XDTM casting technology. Some microstructural and mechanical property information on a mixed α_2/γ intermetallic alloy reinforced with TiB₂ ceramic has been reported by Christodoulou et al (7). Room temperature toughness values of 12-14 MPa (m)^{1/2} were reported for the lamellar structure.

Impact toughness and DBTT's have not been reported for these new IMC's. The present study is to determine the effect of temperature on the impact behavior of two XDTM processed TiB₂ reinforced TiAl IMC's. In order to determine the variation of impact toughness from low to high temperature an existing instrumented tup impact drop tower apparatus had to be modified and new procedures developed to obtain meaningful results.

EXPERIMENTAL DETAILS

Materials

Machined blanks of two near gamma TiB₂ reinforced TiAl IMC materials were obtained. The blanks were unnotched with a 12.5 x 12.5 mm cross section and 63.5 mm length. The compositions were Ti-49 at.% Al + 5 vol. % TiB₂ and Ti-48 at.% Al + 10 vol. % TiB₂, and they were identified as lots S2XD and S3XD, respectively. Both materials were isothermally forged below the transformation temperature at the $(\alpha + \gamma)/(\alpha_2 + \gamma)$ boundary shown in figure 1. This phase diagram of the central region of the Ti-Al system is due to McCullough et.al.(8). A representative microstructure is shown in figure 2 from the S2 XD material. TiB₂ particles are uniformly dispersed in the $(\gamma + \alpha_2)/\gamma$ lamellar matrix. The specimens were notched with a low concentration diamond saw. Oil lubricated, multiple and minute cut depth saw passes were made to a final notch depth ratio, a/W, of 0.2.

The notch width was 0.3 mm with the notch tip radius of 0.15 mm. After notching, the specimens were washed thoroughly with acetone and dried in lab air.

Impact Test Apparatus

An existing drop tower with an instrumented load tup was modified for the present study. The apparatus is shown in figure 3. Impact velocity is controlled by drop height, and impact energy by the addition of weight to the tup head carriage. Test velocity for the present study was 1.5 m/s, and the impact energy was 5.8 J. Data acquisition from the instrumented load tup was obtained from an analog to digital interface in a personal computer. The number of data points per test is 1024 load-time pairs with a total recording window adjustable between 5 and 25 milliseconds. Data reduction was accomplished by commercial software;

Details of the test procedures and modifications to the existing drop tower apparatus for high temperature testing are as follows:

a) Specimen Notch Alignment-

Before the impact test, specimens were positioned on the drop tower anvil using a machined positioning jig for centering in the long axis direction and by centering marks on the anvil in the short axis direction.

b) Data Acquisition Timing-

The data acquisition start flag is attached to the tup head carriage which passes through a capacitive sensor and initiates data acquisition, as shown in figure 3. Due to the possible brittle nature of the test materials the time to failure can be very short. Some of the XD samples were found to fracture in less than 0.25 milliseconds. Thus, a barrel micrometer was mounted on the start flag gate to adjust for precise data acquisition start times. The trigger start height was set to initiate data acquisition 0.38 mm before tup head/sample impact.

c) Heating-

Other investigators have attained high temperature testing, by heating the specimens in an external furnace and then transferring the sample to the impact apparatus for the test (9). To evaluate this method for use in the present study a temperature instrumented sample was heated, and it was found that after transferring the sample, temperature decreased at a significant rate before impact occurred. This method, therefore, was determined to be unacceptable. Winsa and Petrsek (9) also used another heating method in which multiple propane torches were used to attain temperatures of 1099°C. The latter method was tried using an oxyacetylene torch with some degree of success except that some materials were reactive to the gas torch. An approach to attain equilibrium high temperatures rapidly was suggested by the work of Hartman et al (10). Thus, four water cooled quartz infrared line heating lamps were utilized as shown in figure 3. These lights were controlled by a potentiometer for heat flux adjustment from zero to 100 percent along with individually controlled powered switches to provide either two or four lamp heating. For all the testing reported herein 4 lamps were used.

As shown in figure 4, temperature versus time tests were performed on a specimen from the S3XD material to determine the time to achieve internal equilibrium temperature required at various power settings. The sample temperature measurements were made using a 0.005 inch diameter chromel/alumel thermocouple inserted into the notch and held in place with zirconia fiber. The zirconia fiber also prevented the thermocouple from being exposed to the direct radiation of the heating lamps. Since the notch width was greater than the thermocouple diameter, no stress should be present from the thermocouple insertion and zirconia insulation inside of the sample notch. For the results reported herein the specimens were heated for five minutes to achieve internal temperature equilibrium.

d) Instrumented Tup Head Heat Protection -

To protect the instrumented tup head from the lower chamber heat (fig. 3), the tup head was placed in a standby position that was a large distance from the upper chamber to avoid convective heating from the lamps. Cooling of the tup head when placed in the ready position was achieved by a stainless steel, overlapping, manually activated, liquid nitrogen cooled "trap door" which provided a thermal barrier between the lower chamber and the tup head. Desired low tup impact velocity required the tup head to be very close to the lower specimen heating chamber. Since the instrumented tup head had no temperature compensation and was designed to operate at room temperature, the temperature in the upper chamber (position of the tup head in the ready position) was manually maintained at 15° to 20°C. This occurs concurrently while the sample is being heated in the lower chamber. When the upper chamber had achieved the proper temperature (i.e., 20°C) the tup head was lowered to the ready position. When the five minute sample heating period was complete, the "trap door" was opened and the tup head released from the ready position to impact the sample. Immediately upon fracture, the tup head was retracted to the standby position and the heating lamps were extinguished.

In order to confirm that the modification on the existing instrumented drop tower system and possible effects of high and low temperature tests on the instrumented tup, aluminum load calibration standards were tested before and after the tests. These tests verified that the instrumented tup load measurement was in compliance after the low and high temperature tests. Also, to check the system's ability to determine increased toughness at high temperatures, 1090 hot rolled (HR) carbon steel Charpy specimens were tested at 20°C, 300°C, 1000°C. The results are shown in figure 5. These results confirm the system's ability to measure rising toughness at high temperature.

RESULTS AND DISCUSSION

The instrumented tup impact test provides a load - time history as shown in figure 6 for room temperature tests of the 82 XD IMC material. The figure illustrates test repeatability, and the various features of a load-time signal. These features are inertial load, as discussed in reference 11, sample vibrational characteristics and the maximum load. The steep drop off in load after maximum load is characteristic of a brittle material. From the maximum load values at room temperature, maximum bending stresses of 472 MPa and 468 MPa were calculated for 82XD and 83XD, respectively.

Impact load-time histories at the various test temperatures from -192°C to 1032°C for the 83XD material are given in figure 7 which shows the inertial load and vibrational characteristics for all temperatures to be similar. The load-time data is integrated by the computer software to provide the total energy absorbed by the specimen from impact to fracture. The trend of the energy absorbed (i.e. toughness) along with the maximum load values versus test temperatures for both the 82XD and 83XD IMC materials are shown in figure 8. Both of these properties are maximum at room temperature and decrease significantly as temperature increases. The two IMC's, one with 5 vol.% dispersed TiB_2 particulate and the other with 10 vol.%, produced very close absorbed energy and maximum load results.

The above trends of decreasing impact toughness with increasing temperature are reflected in the estimated plane-strain fracture toughness, K_{Ic} . The K_{Ic} values at room temperature are $25.4 \text{ MPa(m)}^{1/2}$ for the 82XD IMC and $24.4 \text{ MPa(m)}^{1/2}$ for 83XD. These values decreased to $10.1 \text{ MPa(m)}^{1/2}$ at 1156°C for 82XD and $10.5 \text{ MPa(m)}^{1/2}$ for 83XD at 1032°C . One possible contribution to the decrease in K_{Ic} with increasing temperature might be the unfavorable difference between the matrix and particulate coefficients of thermal expansion(CTE). Comparison of CTE values

versus temperature from references (7) and (12) for the TiB_2 reinforced - TiAl matrix and TiB_2 particulate show the CTE for TiB_2 to be less than the matrix at temperatures from 20 to 1000°C . This characteristic would impose an unfavorable thermal stress in the material as temperature increases with a subsequent decrease in fracture toughness.

Only the 83XD (10 v/o TiB_2) was tested at -192°C . The energy absorbed and maximum load values were less than those at room temperature (i.e., 20°C). Since these properties decreased above room temperatures, it was anticipated that the absorbed energy and maximum load values at -192°C would be slightly higher than those at room temperature. An explanation of the low temperature result is not apparent.

After the impact tests, Rockwell-C hardnesses were measured on the room temperature and the highest temperature test samples. There was no change in the surface hardness after the high temperature tests. The Rockwell-C values were 30 and 35 for the 82XD and 83XD materials, respectively. The higher value for 83XD confirms the higher TiB_2 volume fraction.

Stereo pair scanning electron microscope (SEM) microfractography was performed on the fracture surfaces. As shown in figure 9, as the impact test temperature becomes 760°C and higher, the fracture morphology changes from transgranular cleavage at room temperature to an even lower fracture toughness mechanism, that is, classic intergranular fracture. The SEM fractography, therefore, yields an explanation for the impact toughness results. The intergranular fracture also illustrates the fine grain size achieved in the test materials. An estimate suggests a grain size number in excess of 10.

CONCLUSIONS

Impact toughness and maximum bending stress for 5 and 10 vol.% TiB_2 reinforced XD-TiAl IMCs decreased from room temperature to temperatures as high as 1100°C . An important experimental observation which supports these results is the change from transgranular to intergranular fracture at the 760°C test temperature. Also noted is the possible contribution to decreasing toughness of unfavorable difference in CTE between TiB_2 and the matrix with increasing temperature.

Data repeatability and verification of the ability to observe high temperature brittle-to-ductile-transition behavior established confidence in the instrumented tup drop tower modification and procedures for high temperatures impact toughness testing.

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└ 10μ

Figure 2-Representative Microstructure from 82XD IMC

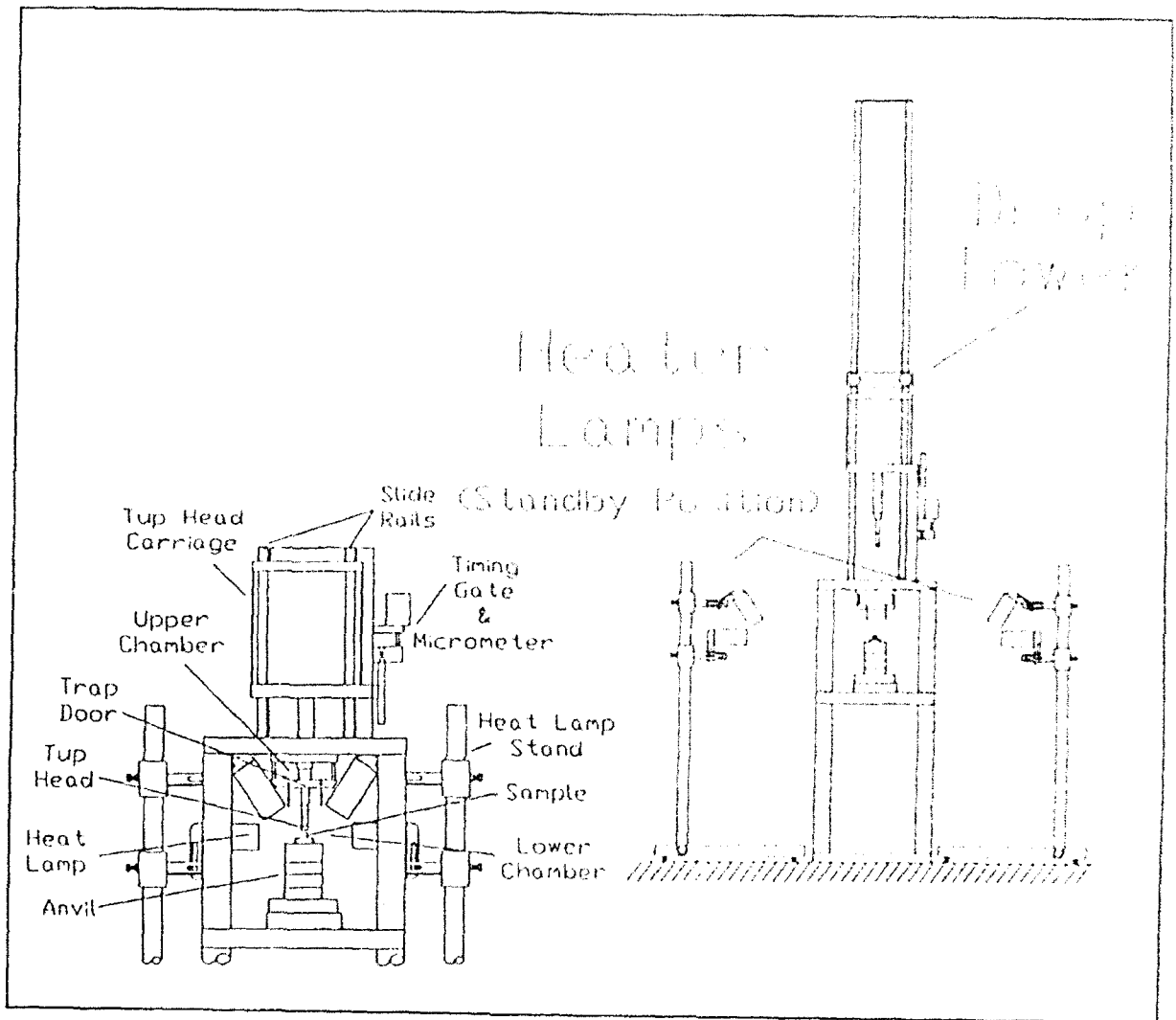
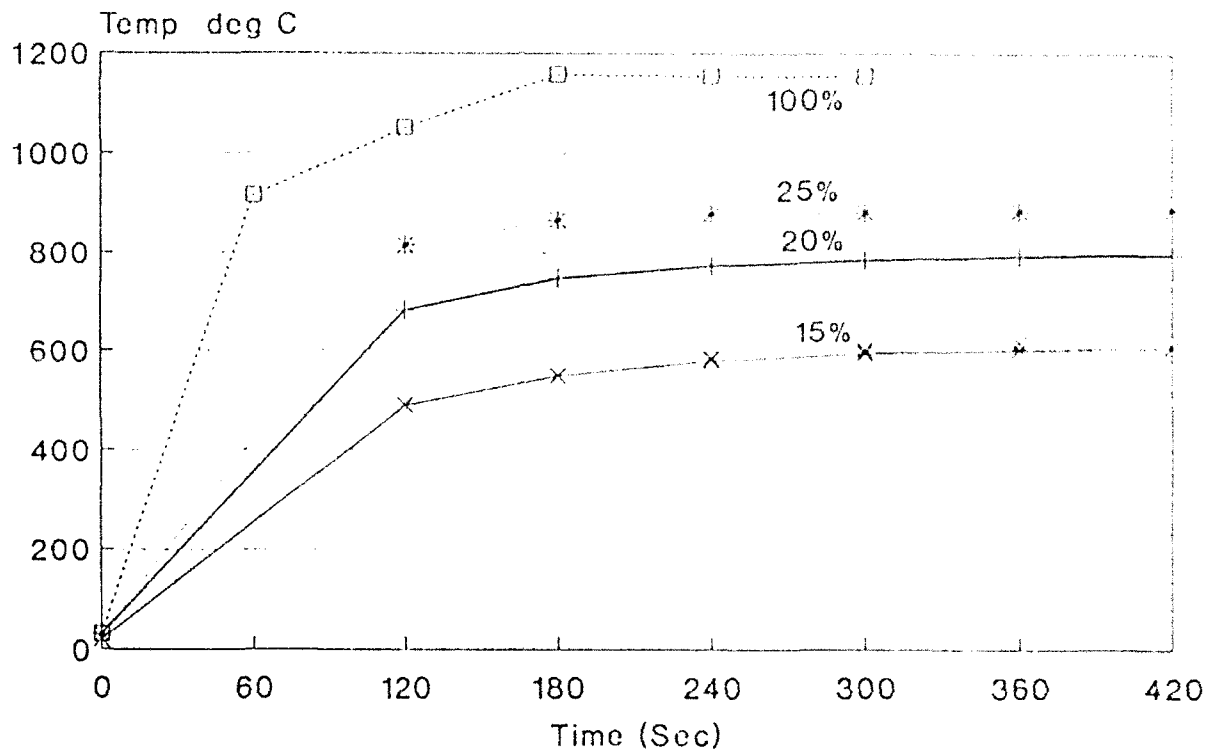


Figure 3-Modified Drop Tower for High Temperature Instrumented Impact Testing

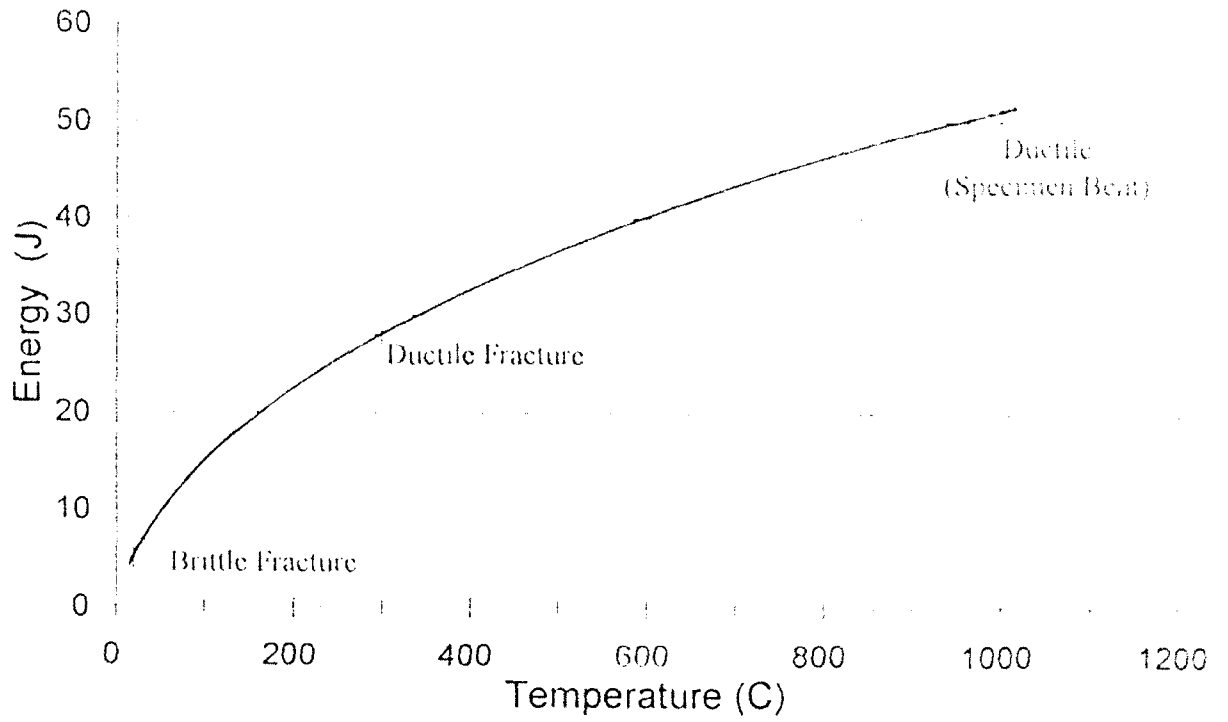
83XD Temperature Profile

Thermocouple in Notch



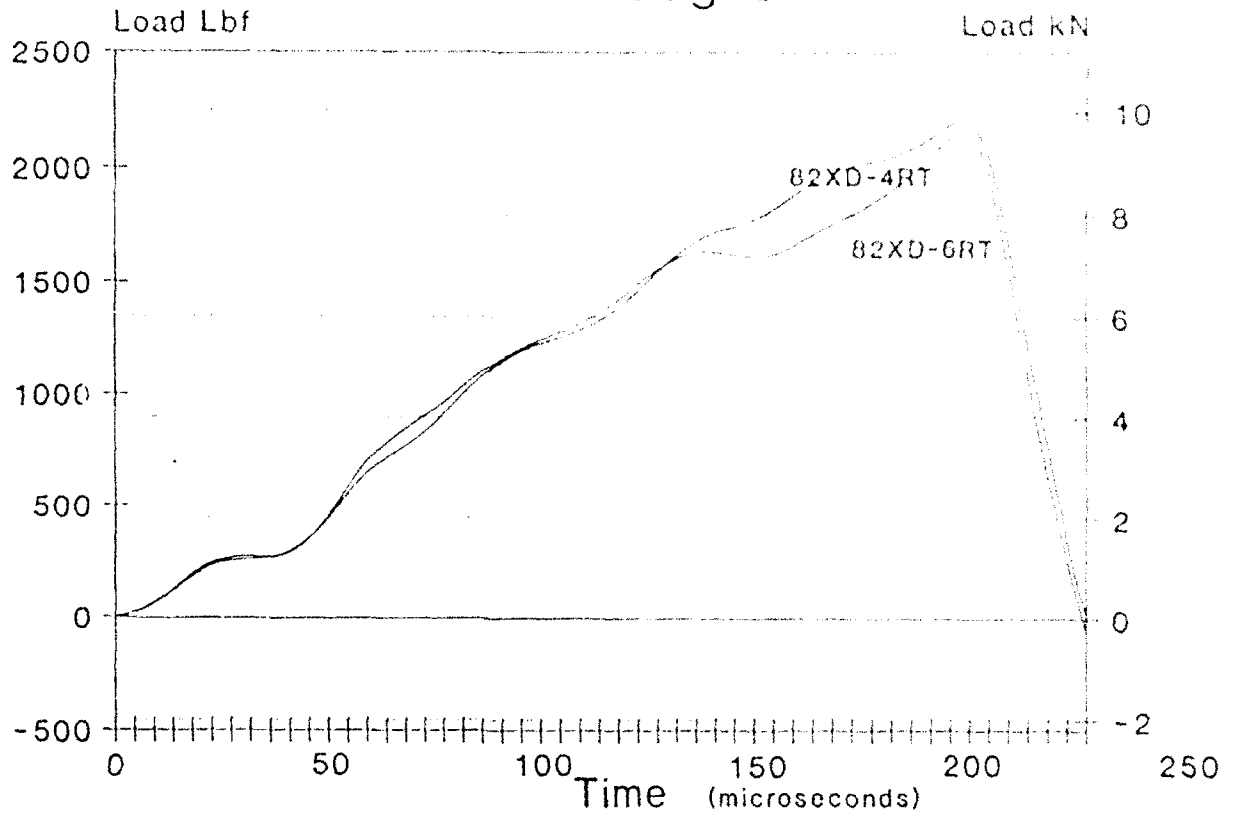
**Figure 4-Temperature-Time Profiles in the 83XD IMC
at Various Power Settings with 4 Heating Lamps**

Impact Energy vs. Temperature 1090 HR Steel

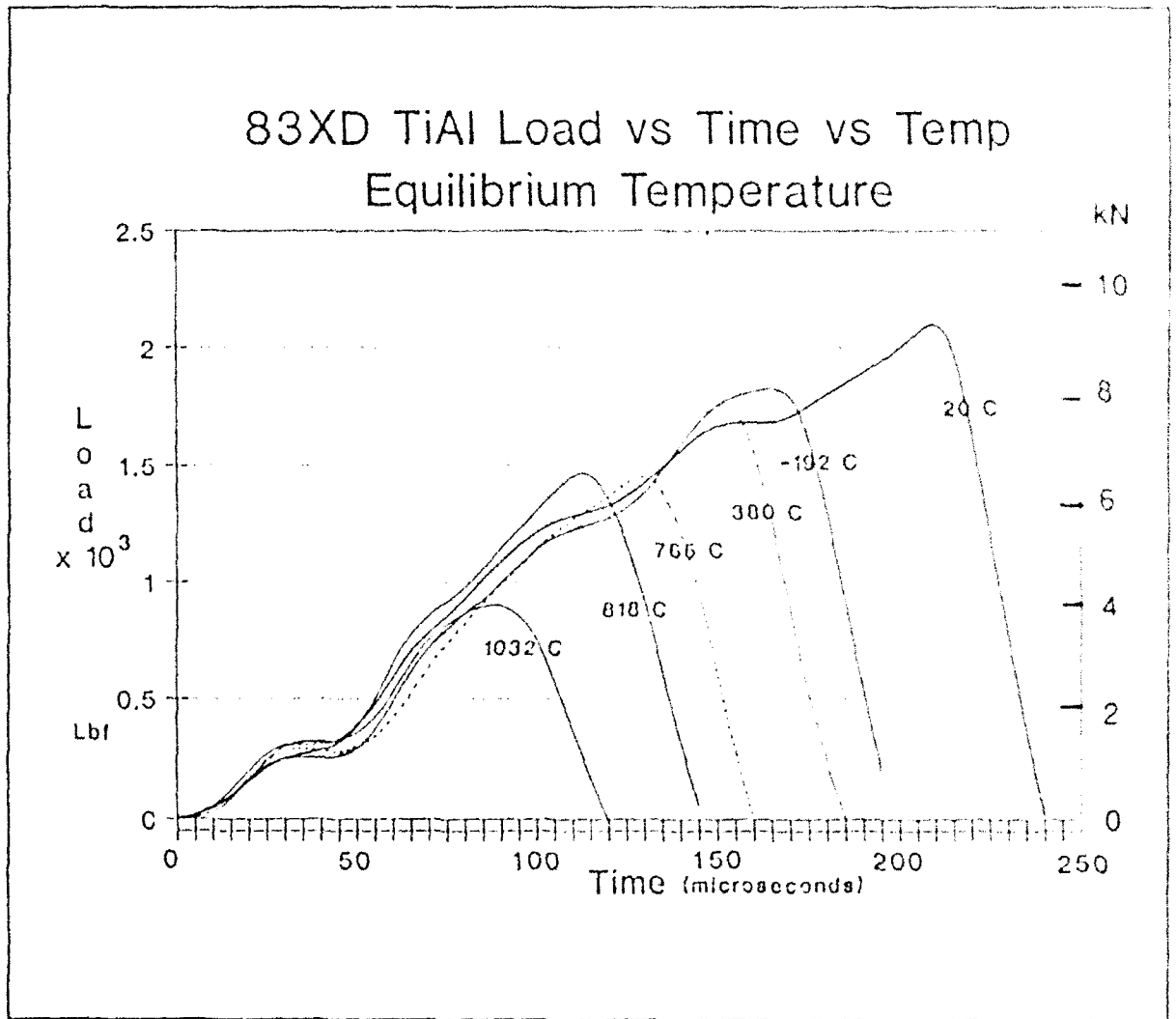


**Figure 5- Impact Fracture Energy versus Temperature
for 1090 HR Steel**

82XD Repeat Comparison 20 deg C



**Figure 6-Repeat Comparison of Load-Time Histories for
82XD IMC Specimens Tested at Room Temperature**



**Figure 7—Load-Time Histories for 83XD IMC Specimens
Tested at Various Temperatures**

82 & 83XD Max Load & Total Energy VS Temperature

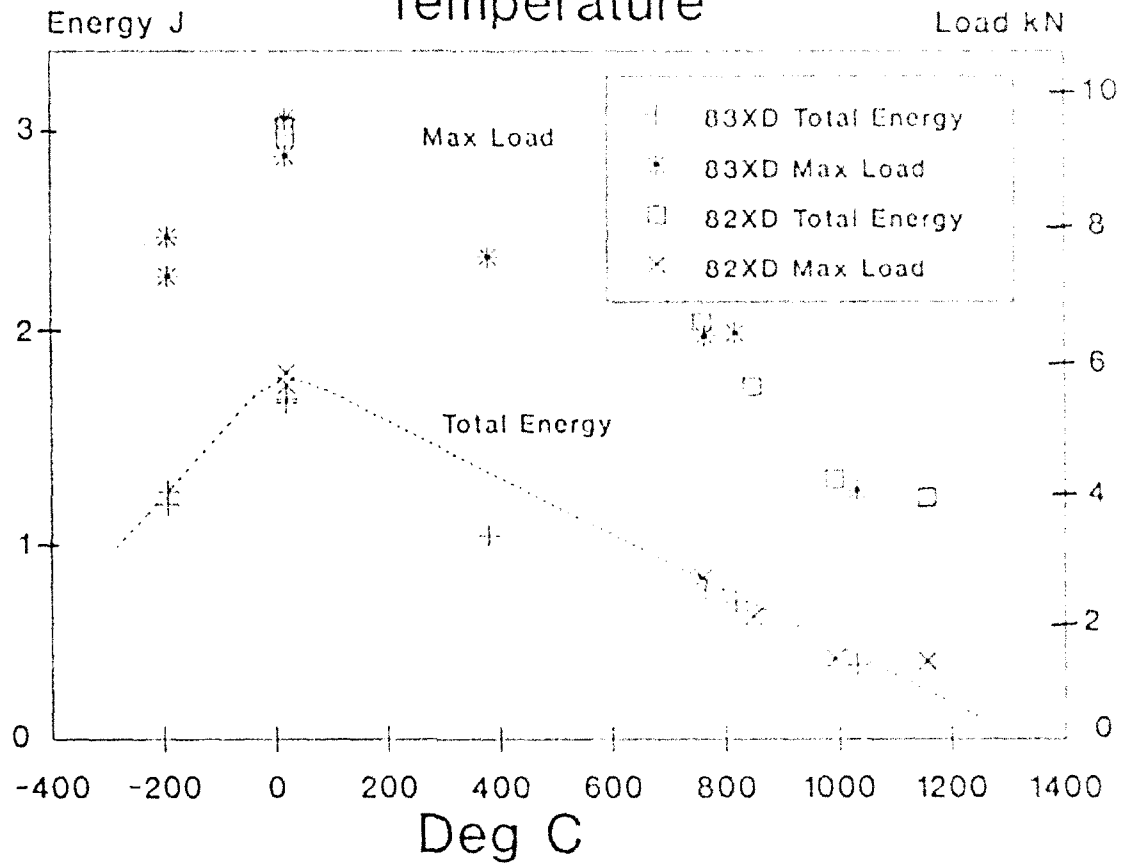
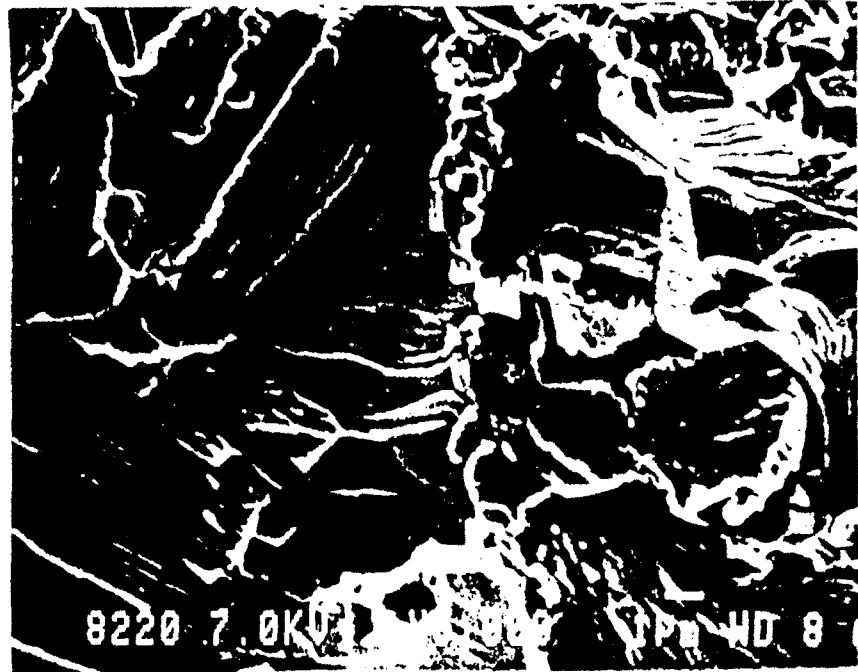
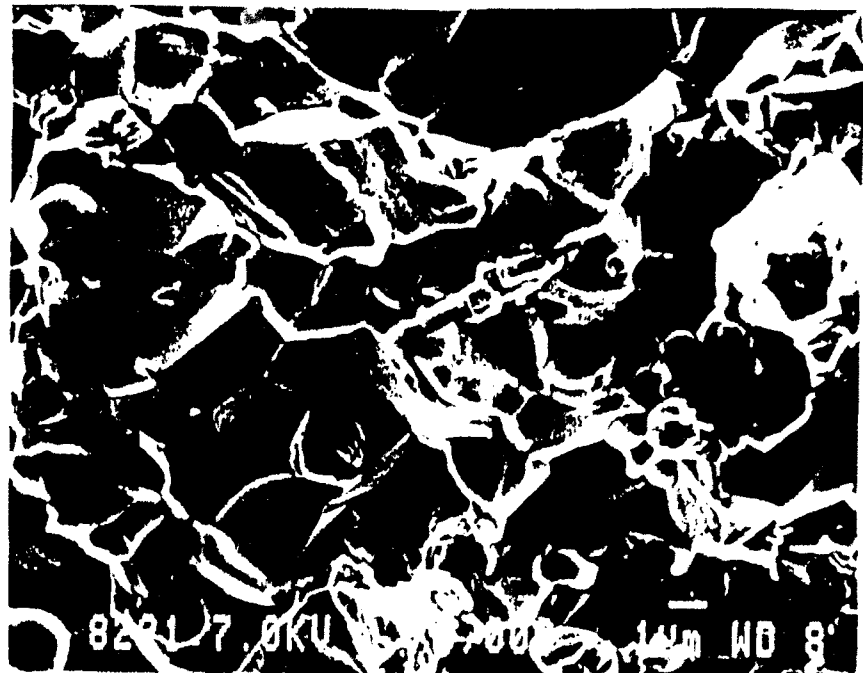


Figure 8-Maximum Absorbed Energy and Load versus Temperature for 82XD and 83XD IMC Materials



(a)



(b)

Figure 9-SEM Fractographs of 82XD IMC Material Tested at (a) 20^oC and (b) 760^oC