Round Robin Comparison of Tensile Results on GlidCop Al25 — D. J. Edwards (Pacific Northwest National Laboratory), S. J. Zinkle (Oak Ridge National Laboratory), S. A. Fabritsiev (DV Efremov Institute), and A. S. Pokrovsky (Research Institute of Atomic Reactors)

SUMMARY

A round robin comparison of the tensile properties of GlidCop™ Al25 oxide dispersion strengthened copper was initiated between collaborating laboratories to evaluate the test and analysis procedures used in the irradiation experiments in SRIAR in Dimitrovgrad. The tests were conducted using the same tensile specimen geometry as used in previous irradiation experiments, with tests at each laboratory being conducted in air or vacuum at 25, 150, and 300°C at a strain rate of $3 \times 10^{-4}\ s^{-1}$. The strength of the GlidCop™ Al25 decreased as a the test temperature increased, with no observable effect of testing in air versus vacuum on the yield and ultimate strengths. The uniform elongation decreased by almost a factor of 3 when the test temperature was raised from room temperature to 300°C, but the total elongation remained roughly constant over the range of test temperatures. Any effect of testing in air on the ductility may have been masked by the scatter introduced into the results because each laboratory tested the specimens in a different grip setup. In light of this, the results of the round robin tests demonstrated that the test and analysis procedures produced essentially the same values for tensile yield and ultimate, but significant variability was present in both the uniform and total elongation measurements due to the gripping technique.

Introduction

A series of irradiation experiments on copper alloys has been conducted in the SM 2-3 reactor in Dimitrovgrad, Russia [1-5]. These experiments have provided considerable data on the tensile properties of the various candidate alloys being considered for use in the ITER device. However, difficulties arose in the testing and analysis that raised questions concerning the accuracy of the data obtained from the tensile tests. Variables such as microstructural variations within the alloy plate, dimensional variations introduced during specimen fabrication, and temperature control during irradiation and testing have all been considered as possible explanations for the apparent differences. Although some of these concerns have been explained by subsequent testing, a series of round robin tests using specimens from one source were initiated to ensure that all of the laboratories involved in the irradiation experiments test the specimens and analyze the data in a consistent manner. It is hoped that these tests will allay any doubts about using the data generated in the SM2-3 irradiation experiments.

Experimental Procedure

The material used in this comparison was GlidCop™ Al25 (ITER Grade 0) supplied to the Efremov Institute by OMG Americas. Type STS tensile specimens were machined from the 20 mm plate and sent to each of the following four laboratories: Pacific Northwest National Laboratory, Oak Ridge National Laboratory, DV Efremov Institute, St. Petersburg, Russia, and the Scientific Research Institute of Atomic Reactors (SRIAR) in Dimitrovgrad, Russia, where the irradiation experiments were conducted. A schematic of the STS tensile geometry is provided in Figure 1. This geometry was also used in the SM2.1 and SM2.3 irradiation experiments on copper alloys.

The tensile tests were conducted in vacuum at ORNL and SRIAR and in open air at PNNL and Efremov. The room temperature tensile tests at ORNL were conducted in either vacuum or open air for purposes of comparison, but all tests at 150 and 300°C were conducted under a vacuum of less than $\sim 10^{-6}$ torr. A strain rate of $\sim 3 \times 10^{-4}\ s^{-1}$ was used for the tests at PNNL, ORNL, and the Efremov Institute, but for the screw driven machines located in the hot cells at SRIAR a slightly higher strain rate of $4.2 \times 10^{-4}\ s^{-1}$ was used. This slight difference in strain rate is known from previous work [6,7] to not have any discernible effect on the tensile properties of the Al25.
In the testing performed at PNNL the specimens were held using specially designed grips that used small pins to align the specimens. The 3.2 mm diameter pinholes were machined into the

specimens after being shipped to PNNL. A face plate screwed onto the front surface of the specimens provided the clamping force necessary to hold the specimens with some support from the pins. Because of alignment difficulties it was determined that the screws needed to be tightened while the entire assembly was under a preload of ~50 lbs. (well below the yield), otherwise a slight misorientation of the specimen or grips would alter the slope of the tensile curve as the specimen and grips realigned themselves at higher loads. Temperature control during the testing was accomplished using a thermocouple attached directly to the exposed grip or gage. Since the tests were conducted in open air, the hold time to equalize the temperature throughout the specimen was only 5 minutes, which also served to minimize the oxidation that occurred at the higher test temperatures.

The tests at ORNL were performed using a modified type SS3 tensile specimen grip system that relied on pins to support the entire specimen load during testing. The 2.4 mm diameter pinholes were machined in the specimens after they were shipped to ORNL. Thermocouples were placed in contact with the gage surface during the testing, and a hold time of approximately 15 to 30 minutes was used to equilibrate the temperature. Some of the initial ORNL tests were conducted at slightly lower temperatures than desired due to a unforeseen problem when the temperature was measured by a single thermocouple placed on the specimen gage region. The grips were colder than the center of the gage section and completely shielded the shoulder and end tab regions of the specimen from the clam-shell furnace. This caused the specimen temperature to be considerably lower than the surrounding furnace temperature even for hold times of more than 30 minutes. Measurements with a second thermocouple placed on the gage section indicated that the actual specimen temperature was about ~30°C lower than measured by the original thermocouple (the original thermocouple was sensing an average of the "colder" specimen and surrounding "hotter" furnace environment temperature). After the discrepancy was found, all subsequent tests were conducted using the temperature reading from the second thermocouple located on the gage section. The tests at the lower temperatures are also included in this report.

The testing conducted in the two Russian laboratories used a "shoulder-loaded" gripping system were 4 pins make contact with the shoulders of the tab end of the specimens, thereby providing the support during testing. The temperature was controlled by thermocouples placed on the gage section of the specimens. The tests were performed in vacuum at SRIAR and in open air at Efremov.

All four laboratories used either a graphical analysis of chart recorder data or data acquired on a computerized data acquisition system to determine the tensile properties. The crosshead displacement was used to measure the actual elongations, not extensometers. The 0.2% yield strength, ultimate strength, uniform and total elongation are all summarized in this report.

Results and Discussion

The yield strengths as measured at all four laboratories as a function of temperature are presented in Figure 2. The yield strength of the GlidCop™ Al25 clearly decreases as the test temperature is increased from 25 to 300°C. The ultimate tensile strength presented in Figure 3 shows basically the same trend, with a scatter band similar to that of the yield strength. The UTS data from U.S. laboratories was systematically higher than the corresponding UTS data from the Efremov Institute and SRIAR, but still within an acceptable error of ±20 MPa. In general each set of strength data is indistinguishable from each other within the scatter of the data, indicating that the yield and ultimate strengths are being consistently and accurately measured in an identical manner across the different laboratories. The influence of test environment (air vs. vacuum) is not evident in the strength data, which is in reasonable agreement with earlier results on the strain rate and temperature dependence of the GlidCop™ alloys [6,7]. The grip system also does not appear to cause any noticeable discrepancies in the measured strength of the specimens.
The measurement of the elongation proved to be a different matter however, as can be seen in Figures 4 and 5. Both the uniform and total elongation measured at PNNL and ORNL are consistently higher than that measured by the Efremov Institute, and the uniform elongation is

typically higher than that measured at SRIAR. Despite this all of the data sets exhibit the same basic trend for the uniform and total elongation. The uniform elongation monotonically decreased with increased with increasing test temperature to a level of ~4% at 300°C. The total elongation was observed to be essentially independent of test temperature, although considerable scatter was present between the data sets from the four laboratories.

Some variability in the uniform elongation may be expected since the relatively low strain hardening capacity of the extruded GlidCop™ plate produces a rather wide plateau in the tensile curve neat the ultimate stress. Therefore, it is somewhat subjective to determine the precise location of where the specimen begins to neck. The differences in the total elongation are harder to explain, even taking into account the large degree of scatter. A pin-loaded grip system might produce higher elongations than either the clamped or shoulder-loaded grip systems since shear stresses associated with misalignment are minimized. However, at present the authors have no explanation for why the Efremov data is consistently lower than that measured at the other three laboratories. The large scatter in the total elongation indicates that the final failure of the specimens is subject to some variability, and may in fact be due to some local inhomogeneity in the microstructure that influences the failure of the specimens. It is interesting to note that the scatter band for the uniform elongation is only ±2%, whereas the data for the total elongation can vary by as much as a factor of two when comparing the data from different laboratories at the same test temperature.

Conclusions

Overall the round robin results show that the tensile strength is reliable and consistently measured by each laboratory. Unforeseen problems with the temperature control and other external variables can of course affect the final results, but if one assumes that those problems do not exist, then the strength data is consistent between the four laboratories. The differences in uniform elongation are minor and don't necessarily change the conclusions that would be drawn from the data, but the large scatter in the total elongation is certainly an issue that needs to be investigated further.

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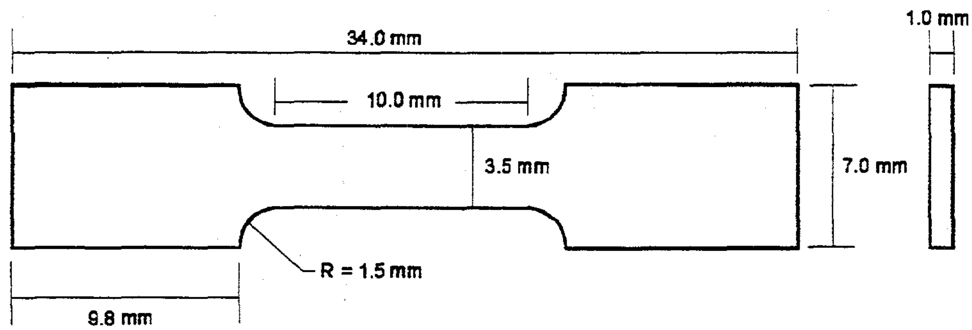

Figure 1 STS tensile geometry used in the round robin comparison. This same geometry was used in the first two series of irradiation experiments (SM2.1 and SM2.3).

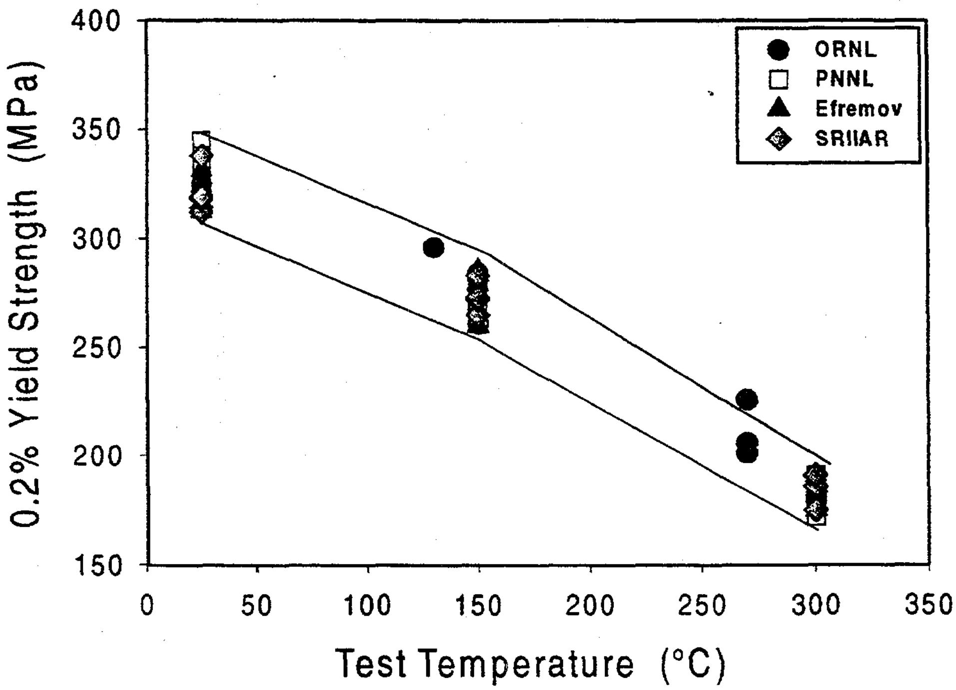

Figure 2 The 0.2% yield strength data from each of the four laboratories is plotted as a function of temperature. The data are all in agreement and demonstrate that the yield strength can be determined consistently.

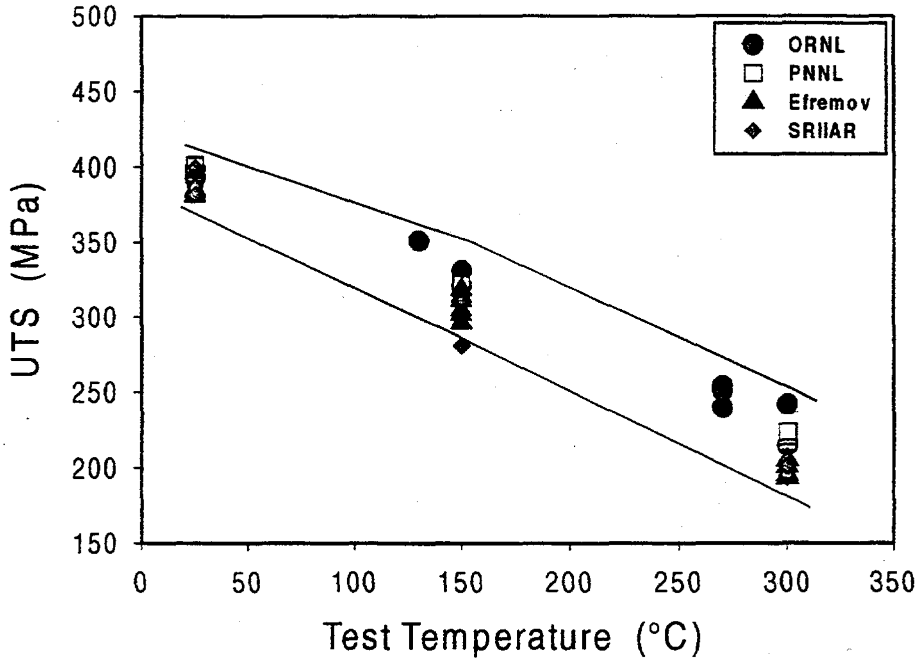

Figure 3 The ultimate strength exhibits more scatter between the four laboratories compared to the yield strength. The data from PNNL and ORNL appear to be systematically higher, but within the overall error of the measurements.

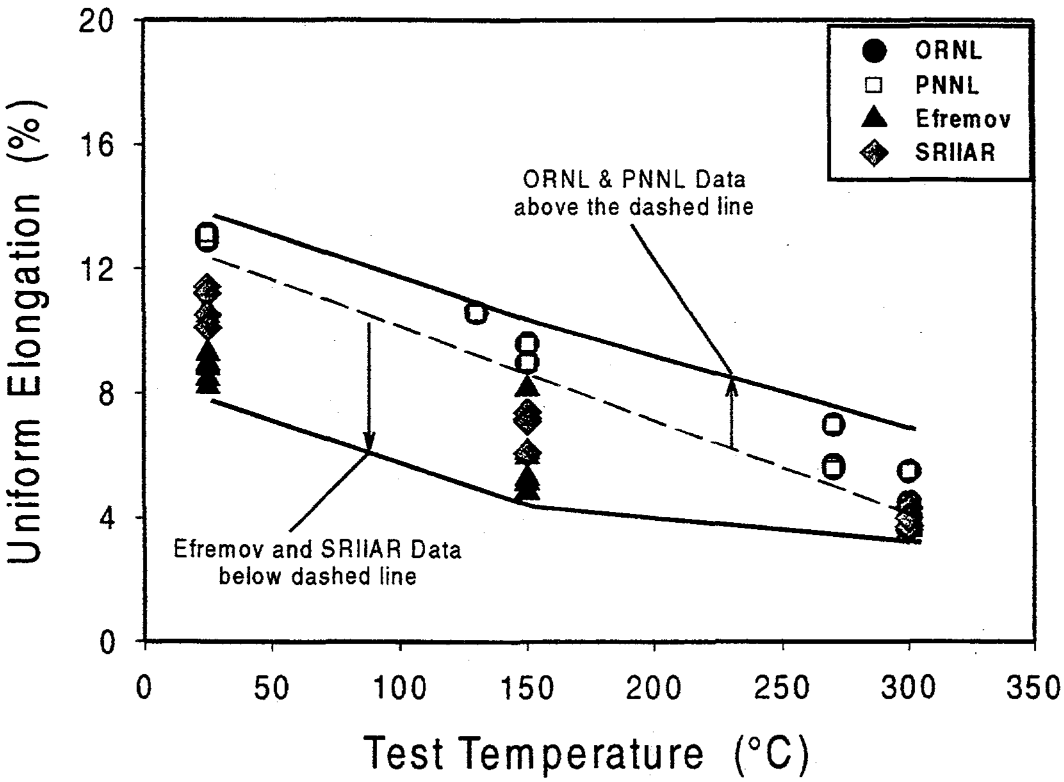

Figure 4 The uniform elongation results from Efremov and SRIAR are both lower on average than the corresponding PNNL and ORNL, but all the data show the same behavior. The manner in which the uniform elongation is chosen off the tensile curve could account for the differences, as well as possible differences introduced by the different gripping systems used. The scatter in the data are about 4-5% at room temperature and 150ºC, with the scatter lower at 300ºC.

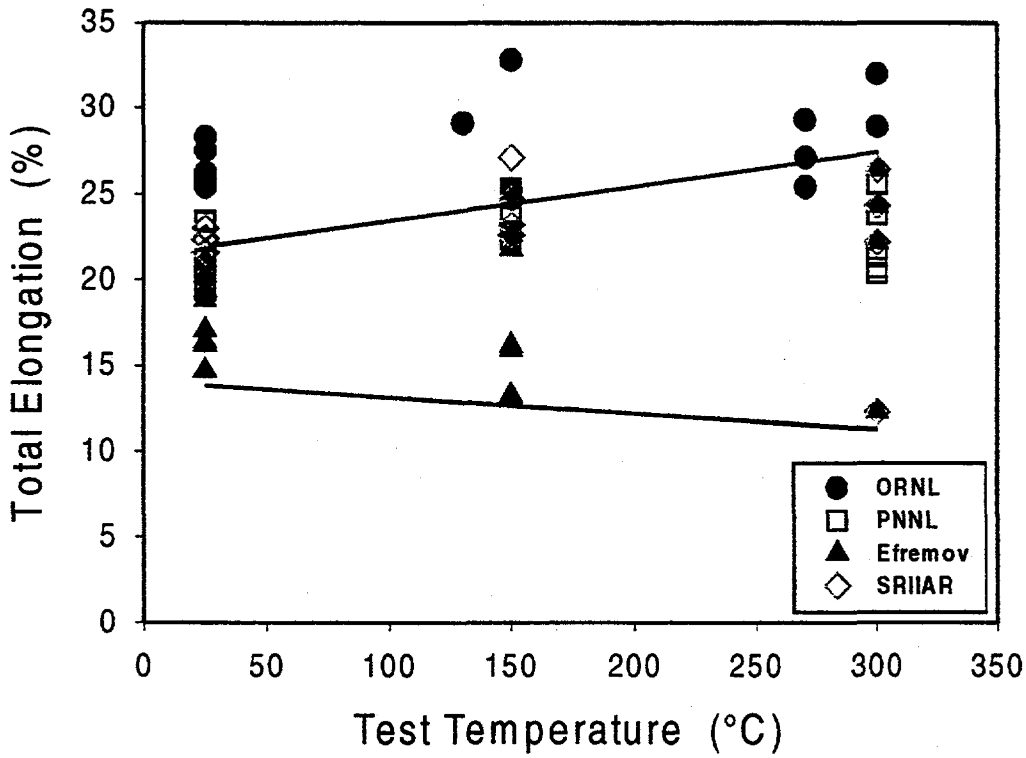

Figure 5 The total elongation data proved to exhibit the most scatter of any of the data sets between the four laboratories. The trend lines are drawn to reflect the Efremov data set only. It remains uncertain as to why the total elongation measured by the Efremov Institute exhibits greater scatter and lower values on average than that measured by the other three laboratories.