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On the High Temperature Testing of Superplastic Materials

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The simplest test in characterizing the behavior of superplastic materials is the uniaxial tensile test. Since superplasticity is achieved at relatively high temperatures, heat involvement adds so many unpredictable problems to the simplest testing technique. In spite of the vast number of research activities directed towards studying the various aspects of superplastic deformation, there is a lack of a standardized testing procedure that can tackle the various issues associated with high temperature testing. In this work, we attempt to shed some light on the controversial issues associated with high temperature superplastic testing. The effects of various testing procedures and parameters on the accuracy of the results are investigated. We address the issues related to gripping and test sample geometry, heat and temperature effects, and comment on the available testing and analysis procedures. We hope that this study highlights the urgent need to develop a standardized testing approach that takes into account all the important issues affecting high temperature testing.

Keywords heating and holding times, high temperature uniaxial tensile testing, sample geometry and gripping, superplastic testing

1. Introduction

Uniaxial tensile testing is the most common and the easiest testing procedure for characterizing the mechanical behavior of different materials. This simplicity is reflected by the worldwide standardization of its aspects: mainly specimen's geometry selection and stress/strain measurements. However, this is only true at room temperature. This simplicity turns into a hard-to-ignore ambiguity when heat becomes involved. There are many issues that are not important in room-temperature (RT) testing that become unavoidably crucial in high-temperature (HT) testing. Some of these issues are

1. Specimen's geometry
2. Grip's design and gripping method
3. Strain measurement
4. Load measurement
5. Thermal expansion
6. Heating time

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The available published studies do not provide guidelines on how to account for these issues when testing superplastic material. Although, some investigators may have used specialized and custom made testing set-ups which account for these issues, the details of conducting the tests are generally not reported. This paper does not question the accuracy of reported experimental data. The objective of this report is to highlight the need for developing standards for testing superplastic materials. This is especially important if a comprehensive database for superplastic materials is to be developed.

The ASTM E21 sets the standard methods for high temperature tensile testing of metallic materials (Ref 1). Since superplastic testing is usually carried out at elevated temperatures, E21 offers some guidelines regarding testing apparatus, specimens, stress/strain measurements, temperature measurement, calibration, and testing procedure. However, some of the procedure-related issues are not fully explained such as thermal expansion of the specimen, holding time before straining, and the mechanism by which gripping-heating-testing are combined. The ASTM E21 is a high temperature tensile testing standard and cannot be expected to tackle the specific issues of superplastic testing.

The Japanese Industrial Standard JIS H7501 proposed a procedure for evaluating the tensile properties of metallic superplastic metals in 2002 (Ref 2). It was the first attempt to develop standards for testing superplastic materials. However, the report does indeed lack the comprehension needed to cover all the controversial issues associated with the nature of superplastic testing at high temperatures. Some issues including the thermal expansion due to heating, gripping, and load cell sensitivity were not addressed. Other important issues were discussed briefly without setting adequate guidelines to control them, as the case with the time required for heating prior to straining. In addition, some of the guidelines that were selected may be a subject of controversy, for example,

- Characterizing the superplastic region by a minimum of 300% elongation.

- Setting the constant cross head velocity test as the standard way for the application of load.
- Evaluating the strain rate sensitivity index from the logarithmic stress/strain rate curves, and not strain rate jump tests.

In a previous work on the high temperature deformation characteristics of AZ31 Mg alloy (Ref 3), the issue of gripping the sample was highlighted as a serious problem, which we were able to overcome by altering the mechanism through which gripping action is achieved. A new set of grips were designed, built and then tested over several stages, until all the associated problems have been either eliminated or minimized to a very good extent. It became then of great importance to expand that effort and shed some light on the controversial issues associated with high temperature superplastic testing.

It is important to note that the presented discussion on any issue may not necessarily offer solutions to one or more of the associated problems. It is hoped that this work will motivate further discussions on the issues related to high temperature testing of superplastic materials.

2. Testing Equipment and Experimental Procedure

The equipment used to conduct the uniaxial tensile tests throughout this study is the INSTRON 5582 universal testing machine, equipped with electrical resistance heating chamber (furnace) that provides a maximum temperature of 610 °C, and can maintain a temperature variation of ±1 °C. A ±5 KN load cell was used for load measurement in all the tests. Unless otherwise stated, the uniaxial tensile tests were carried out maintaining a constant strain rate value.

The material used throughout this study is the commercial alloy AZ31B-H24 in the form of 3.22 mm (0.125 in.) thick sheet, with average initial grain size of about 5 microns. Test samples of size 19 × 6 mm were cut along the rolling direction of the as received sheet.

The test sample is fitted between the grips inside the heating chamber, and the 'specimen protect' controller is activated before heating phase is started. This controller induces the cross head beam to move up or down in a way that maintains a very small preset load value (±2.5 N), allowing the specimen to expand without distortion. When the desired temperature is reached, some additional equilibrium time is allowed (to be discussed later), after which the test is started. Stress measurement is directly obtained from the load cell reading. Due to the large Superplastic deformation, the lack of high temperature extensometers, and the relatively short gauge-length samples, strain measurement is established from the direct displacement of the cross head beam.

3. Grips and Gripping Issues

3.1 Old Grips and Test Samples

The machine is equipped with a set of grips designed to stand the high temperatures during testing. Each grip utilizes two sliding wedge-shaped grip inserts that apply pressure on the surface of the test sample, causing the gripping action. The inner surface of each of the two matching grip inserts is knurled to guarantee firm gripping and eliminate slippage. These grips were first used to perform a number of tests under different combinations of temperature and strain rate. But after a number of tests, a status was reached where some problems escalated to the point at which the grips had to be completely redesigned. The most important problems are discussed in details.

3.1.1 Slippage. The mechanism by which the sliding-action grip inserts work seems to be okay at room temperature. But when the testing temperature was raised, the test sample was observed to loose contact with the grip inserts and slip out of the grip. This was reflected by an abrupt drop in the stress-strain curve, in addition to the observed slippage marks on the surface of the test sample, as shown in Fig. 1(a).

The problem was observed to become more serious as the test temperature is raised, due to the thermal softening of the test sample, which caused the gripping pressure to decline down to the point where it is not enough to hold the sample in place. In addition, flow of material out of the grip region into the gauge length region makes the grip region thinner, and escalates the problem.

To minimize the problem, excessive twisting force had to be applied when gripping the sample, which caused another problem indeed.

3.1.2 Imposed Twisting Torque. This problem is caused by the mechanism by which these grips work. The grip handle is twisted for the grip inserts to slide outwards, which consequently squeeze and hold the sample tight in place. But this action imposes some twisting torque on the test sample, which might affect the uniaxiality of the tensile test. In some cases, and trying to avoid the slippage problem, high twisting caused a permanent distortion in the test sample.

3.1.3 Material Flow into the Gauge Length Region. In addition to its effect in reducing the gripping pressure on the sample, as explained before; the fact that the material flows from the grip area into the gauge length area implies a sort of distortion in the strain measurement. This is simply referred to that an unaccounted-for material chunk is contributing to the total deformation measured during the test. This issue becomes

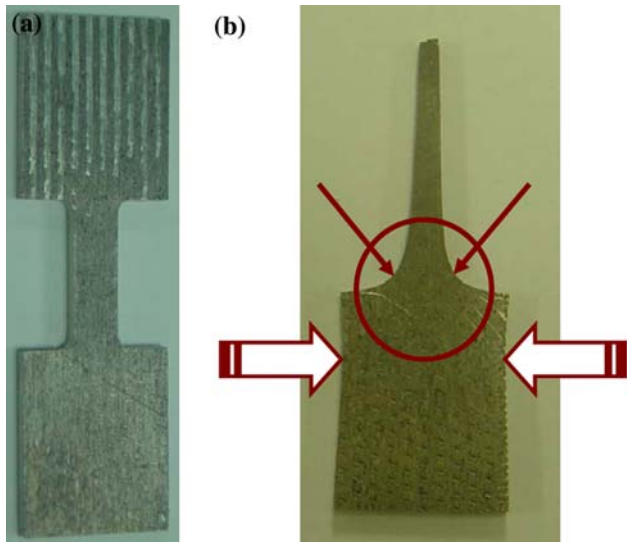

Fig. 1 (a) Slippage marks on the test specimen. (b) Material flow from the grip region into the gauge length region

more observable at higher temperatures, where the material is very soft and less resistive to flow in. Figure 1(b) illustrates how clear and important this issue can be.

3.1.4 Gauge Length Issue. Among all this, the most serious problem was how to define the exact gauge length? The importance of defining the exact gauge length accurately is reflected

- Directly: on the strain measurement
- Indirectly: on the cross head beam controlled speed during a constant strain rate test.

Figure 2 explains the confusion in the gauge length determination, by showing the three possible positions for the test sample with respect to the grip inserts. With these grips, it is almost impossible to guarantee that the edge of the grip insert matches the shoulder of the test sample.

These issues altogether prompted us to modify the sample's geometry and then design and build a new set of grips that minimizes or eliminates the previously highlighted issues.

3.2 Modified Grips and Test Samples

Studying the design of the available grips, and the mechanism by which the uniaxial load is exerted on the specimen during the test, the following remarks are outlined:

- The tensile load has to be exerted on the shoulders of the specimen, and not to be applied through friction between the sample and the grips.
- An alignment pin in the middle of the grip is essentially important.
- The mechanism by which the sample is gripped should eliminate or minimize any imposed non-uniaxial loads, like torsion or bending.
- A restraint is to be provided at the threshold of the gauge length region to minimize material flow during the test.

Based on these remarks, a new set of grips were designed and built, as shown in Fig. 3. As the schematic drawings show, this design eliminates any possibility for slippage, and leaves no question marks about the actual gauge length, since the specimen is being pulled from its shoulders at both ends of the gauge length region. The cover part of each grip is made slid-able over a set of male/female type rails (two side and two front), to assure proper alignment. This also gives the grips the ability to take test samples of any thickness between 0 and 6.35 mm. With these grips, it is not necessary at all to tighten the grips firmly, because tension force is not applied through the grip/sample interface. Bolts here act as alignment pins as well.

The presented set of grips has been used to conduct a series of different tests, in which they have proven to tackle all the problems faced by the original grips, to a large extent.

The following points are considered for the design of the test sample's geometry:

- Relatively short gauge length, to allow large deformation, due to the nature of superplastic tests.
- Minimum corner fillet radius, since the gauge length is measured between the two shoulders of the test specimen.
- Adequate shoulder width.

Currently, researchers use different specimen designs to test Superplastic materials. The geometry of the sample used in the JIS H7501 testing method (Ref 2) is not justified and raise many questions especially regarding the gauge length and the large fillet radius. The ASTM E21 (Ref 1) specimen selection is more convincing, but does not offer a definite geometry when extensometers cannot be used, as it is the case with superplastic testing.

4. Heating-Related Issues

4.1 Protecting the Sample during Heating

If heating is applied when the sample is fixed between the lower and upper parts of the grips, severe compressive loads on the sample may result due to the thermal expansion of the sample and the grips. This may lead to the buckling and

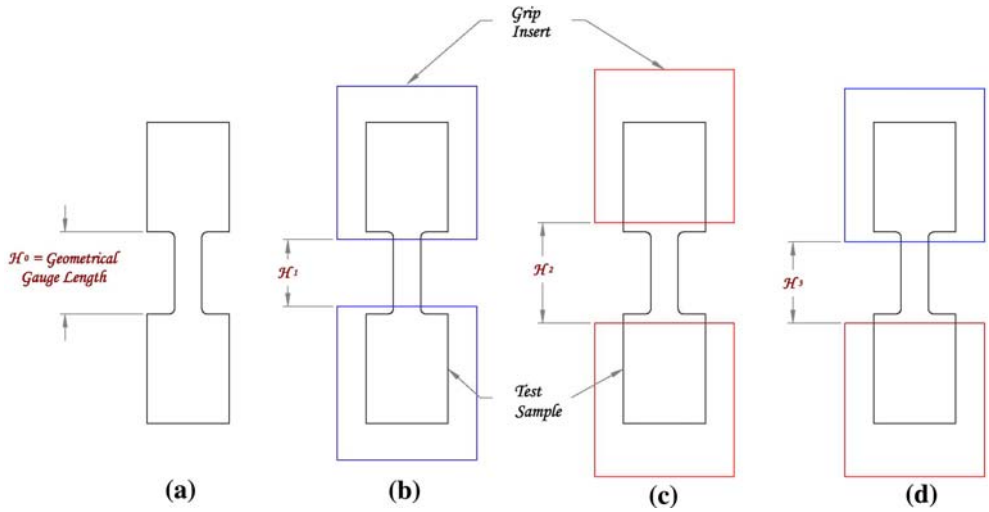

Fig. 2 (a) Geometrically determined gauge length [H_0]. (b) Grip inserts' edges inside the geometrical gauge length [$H_1 < H_0$]. (c) Grip inserts' edges outside the geometrical gauge length [$H_2 > H_0$]. (d) Combination of (b) and (c)

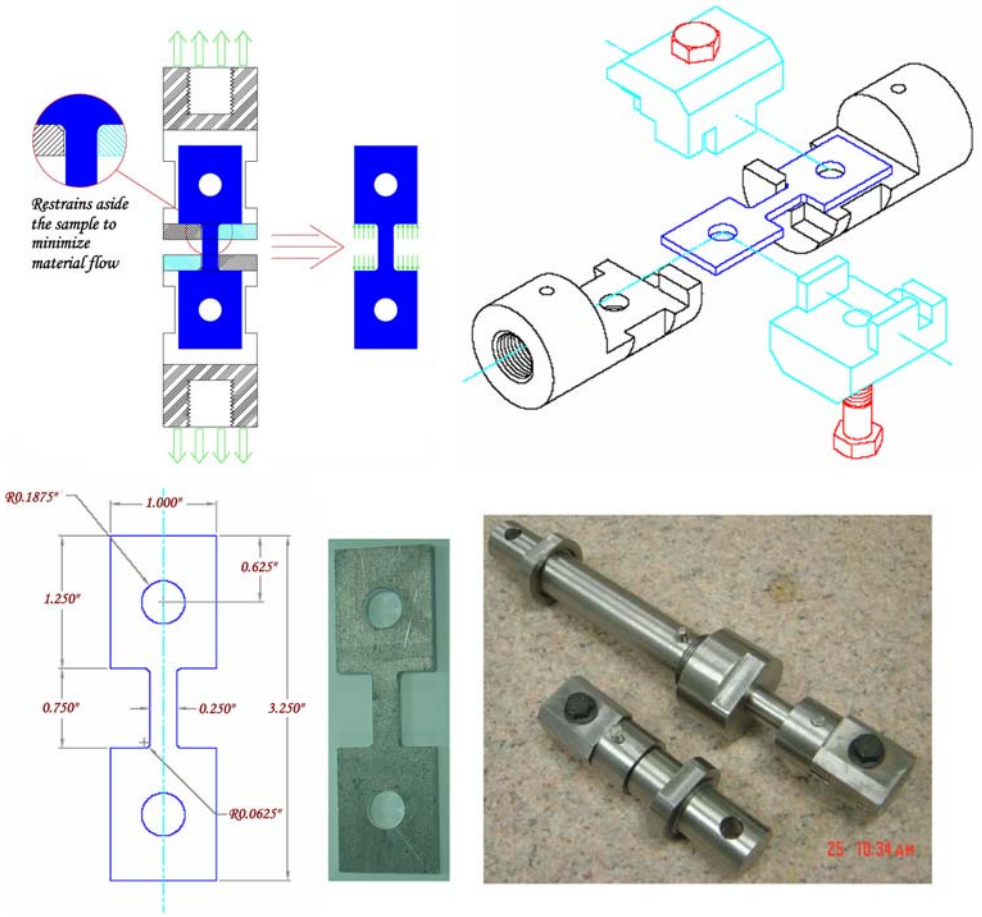

Fig. 3 Design of the new grips and test specimen

distortion of the sample. To avoid this, "protect sample" control option, which is available on most recent universal testing machines, must be applied. This option controls the movement of the cross head beam, to maintain "almost" zero-load on the sample throughout the heating period.

4.2 Thermal Expansion of the Test Sample

The test sample is expected to expand during the heating phase of the test, and the question is: shall the change in the gauge length due to heating be accounted for? For a constant cross head beam velocity test, the gauge length value does not affect the loading path during the test. But for a constant true strain rate test, the velocity of the cross head beam is determined based on the gauge length value.

The mean coefficient of linear thermal expansion for polycrystalline magnesium is about 29.9 μm/m °C, for temperatures in the range from 20 to 500 °C (Ref 4, 5). For a temperature of 500 °C, the maximum thermal expansion the gauge length undergoes was estimated to be 0.27 mm, which is about 1.44% of the original gauge length. Similar estimates were made for other temperatures, as listed in Table 1. In order to determine if these values are small enough to be ignored, two tests were conducted at 375 °C and 5×10^{-4} s^{-1}: in one of the test, the thermal expansion was taken into account and was ignored in the other test. The true stress/strain rate curves were almost identical and the variation in the gauge length of the sample due to thermal expansion can be ignored.

The total distance the cross head beam moved during the heating phase before reaching the thermal equilibrium point (to be explained later) was recorded and averaged about 3.25 mm for a number of tests conducted at 375 °C. For steel, the mean coefficient of linear thermal expansion is about 5/8 of that of magnesium (Ref 5). The ratio of the total length of steel components inside the furnace (grips and connecting shafts) to the magnesium sample's length is about 580/19. Consequently, the ratio of the total expansion in steel to that of the magnesium sample is about 19:1, which leaves the magnesium sample with about 0.17 mm out of the total 3.25 mm.

Moreover, for each test, the actual length of the sample at fracture was measured and compared to the computer reading that corresponds to the cross head beam movement.

Table 1 Percentage change in the gauge length due to the thermal expansion

Temperature, °C	Change, %
250	0.64
300	0.78
350	0.96
400	1.10
450	1.29
500	1.44

The difference was always less than 2%.

In conclusion, the thermal expansion of the specimen is small enough and can be ignored.

In addition, the extra length the sample gains as a result of heating compensates the reduced effective gauge length due to the small fillets at both ends.

4.3 Effect of Heating Time on Stress-Strain Curves

It was described earlier in the testing procedure that the test sample is griped and then heated till the desired temperature is reached, after which a certain time is allowed to reach thermal equilibrium. Two important questions arise. How long does it take to reach the state of thermal equilibrium, and how do we define thermal equilibrium?

In the following discussion, the total heating time is defined as the period from the point when heating starts, until tensioning is started. The holding time is defined as the time from reaching the desired test temperature until tensioning is started. The total heating time is the sum of the time it takes the furnace to reach the desired test temperature and the holding time.

Some investigators alluded to this issue in different ways. Tan and Tan (Ref 6) heated the samples to the desired test temperature, followed by 20-min holding time to ensure thermal equilibrium, for tests in the range of 250–400 °C. Wu et al. (Ref 7) tested the material between 150–500 °C, and mentioned the allowance of 20 min for stabilizing prior to testing. Mohri et al. (Ref 8) carried out their tests at 300 °C, where the specimens required 1800 s to equilibrate prior to the initiation of straining. Chino and Iwasaki (Ref 9) also equilibrated the samples for 1800 s, yet they tested the material between 300 and 450 °C. For the tests conducted by Jäger et al. (Ref 10) at temperatures ranging between RT and 400 °C, each specimen was tempered for 30 minutes. Lee et al. (Ref 11) specified a short period of 60 s holding-time before straining, for tests ranging between 250 and 500 °C. Kim et al. (Ref 12) on the other hand, followed a different heating route in their tests between 300 and 410 °C. They heated the tensile jig inside the furnace, and then inserted the test sample into the heated jig and held it for 10 min before starting the test.

From the above review, the following is noted:

- The different researchers allowed different holding times to achieve thermal equilibrium in the test sample.
- Investigators who conducted their tests at different temperatures used the same holding time for all temperatures to reach thermal equilibrium.
- None of the available studies provided an explanation on how to determine the necessary holding time, how to practically define thermal equilibrium and why it is important to reach thermal equilibrium.

The available discussions on heating/holding time in the literature fail to present a practical guide to address this critical issue. ASTM standards E21 requires a holding time of not less than 20 min without any reference to the material or the test temperature (Ref 1). The JIS left the selection of the heating time for the interested parties to agree on provided that uniform temperature distribution is assured (Ref 2).

In the following sections, we investigate this important issue and present our approach to address it. Several uniaxial tensile tests at different combinations of temperatures and strain rates were repeated at the exact conditions, except for the holding time.

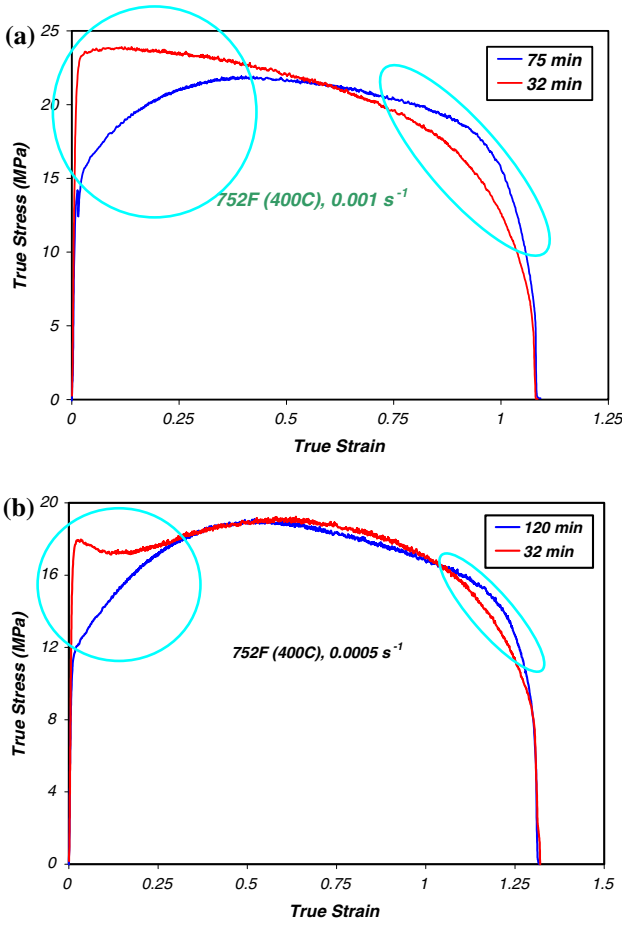

Fig. 4 Effect of total heating time on the resulting stress-strain curves

The effects of holding time on the true stress-strain curves for two strain rates at 400 °C are shown in Fig. 4. The holding time has a significant effect on the flow stress, and similar behavior was also observed at other temperatures. The differences are particularly significant during the early stages of deformation, where flow stress measurement (for a particular strain rate) is usually taken to construct the sigmoidal-shaped stress/strain rate curve. This effect can be explained by two main points:

- Microstructural changes in the sample as a result of maintaining high temperature for long durations. This may lead to significant static grain growth. Independent studies are needed to assess the level of static grain growth and other microstructural changes that may result due to extended periods of heating. For the AZ31 alloy considered here, preliminary results show that this alloy undergoes static grain growth as a result of increasing the holding time. Figure 5 compares the microstructures of three samples subjected to different heating times at 375 °C: as received (zero heating time), 60 min total heating time, and 400 min total heating time. The average grain size has increased from around 5 microns to about 7.5 microns after 60 min of heating and to about 8.5 microns after 400 min of heating. More detailed study of microstructural changes due to holding time is needed and is currently being investigated.
- When imposing a certain strain rate before equilibrium state is reached, thermal expansion of the setup counteracts

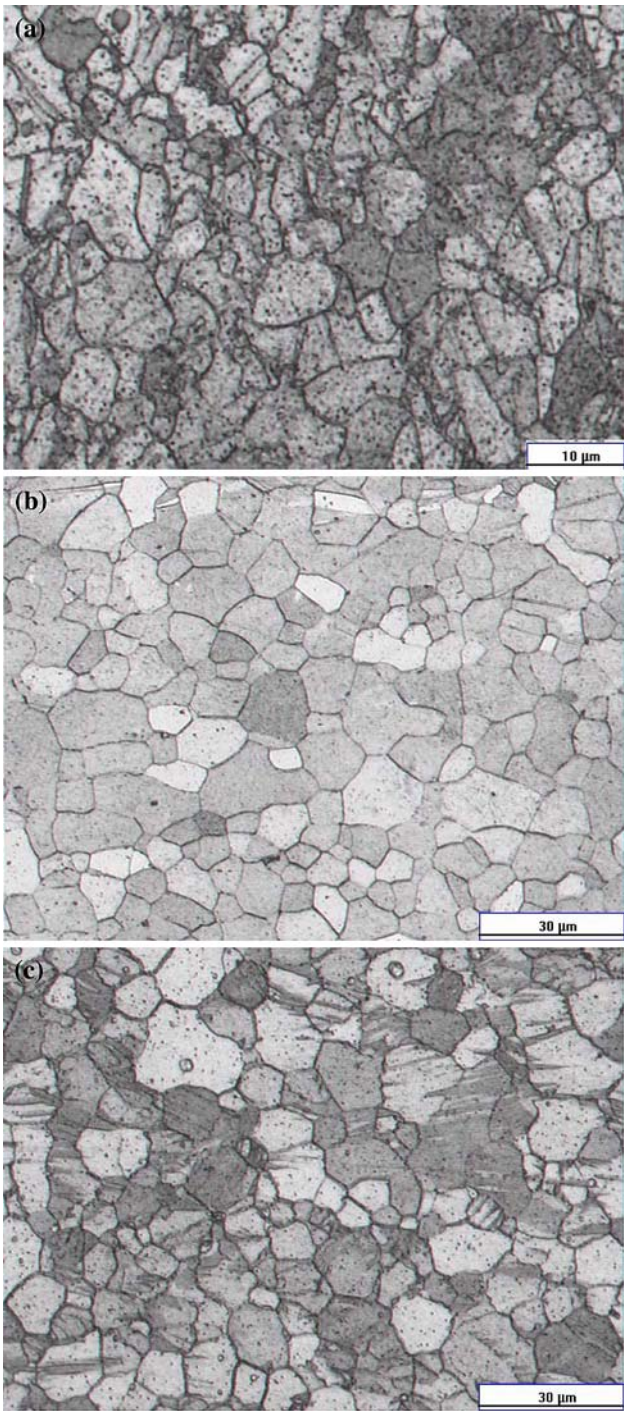

Fig. 5 Grain structure for samples heated for different times at 375 °C. (a) As received (b) 60 min (c) 400 min

the imposed strain rate reducing its effective value. This issue is critical especially at low strain rates.

The accuracy of the sigmoidal-shaped stress/strain rate curve is strongly affected by the holding time. Non-realistic strain rate sensitivity values were obtained from a stress/strain rate curve constructed at 400 °C from tests conducted with no holding time (i.e. before reaching thermal equilibrium). Allowing enough holding is important to ensure uniform temperature distribution in the test specimen.

The furnace we used provides a maximum heating rate of about 25 °C/min, which decreases gradually to about 2 °C/min at the end of the heating stage. Such a heating rate should guarantee a homogenous temperature distribution in few minutes after reaching the desired test's temperature, due to the small size of the test sample. On the other hand, the steel components inside the furnace (grips and connecting shafts) require longer time to acquire temperature homogeneity. And until that state is reached, the cross head beam will keep moving to accommodate the thermal expansion of the steel components, and protect the test sample. The seriousness of this issue was detected in the very low strain rate tests. When such tests were started with no holding time, the load cell reading indicated compressive loading on the test sample, simply because the imposed cross head speed is smaller than the rate at which the steel bulk is expanding.

Therefore, we propose to define the necessary holding time to reach thermal equilibrium as the time needed for the cross head beam to stop moving, indicating that thermal expansion is almost ceased. Following this definition, the required total heating time to reach thermal equilibrium for different test temperatures were measured and summarized in Table 2. It is important to note that the necessary holding time varies with the desired test temperature.

4.4 Effect of Temperature on the Load Cell Reading

Although the effect of test temperature on the load cell reading may not seem to be relevant, however, it was a serious problem simply because it was 'unexpected'. The problem was discovered during the low strain-rate tests, which last for long times. It was observed that after fracture takes place, and the two parts of the broken sample are entirely apart, the load cell reading was not zero. In fact, in many occasions, the load reading after fracture was more than 10 N, and in one case even reached 29 N. For a test conducted at 10^{-4} s^{-1} and 400 °C, a 10 N force is equivalent to a 0.5 MPa true stress in the early stage of deformation (~18% of the flow stress), and about 2.65 MPa true stress in the very last stage of deformation (~48% of the flow stress at that point). Figure 6 shows the true stress/strain curve obtained from a uniaxial tensile test at 375 °C and 10^{-5} s^{-1}. When the sample fractured, the load cell reading was still 6.2 N, equivalent to 4.3 MPa or approximately 45% of the flow stress just before fracture.

To investigate this problem, a test was prepared as usual, with the exception that no test sample was used; the grips were in position inside the furnace yet apart. Testing temperature was set to 500 °C, and the applied strain rate was set to zero. The test started along with heating. Since there is no sample between the lower and upper grips, one should expect the load cell reading to stay zero during the test. Surprisingly, the load reading kept increasing gradually, and a maximum value of 58 N was recorded! It became clear that the load cell is experiencing some heat that alters its reading. This was unexpected simply because the furnace is made by the same manufacturer who built the testing machine and it was intended for use with the same tensile testing machine.

To solve the problem, the gap between the shaft and the hole on the upper surface of the furnace was minimized by means of an insulating material. In addition, a fan was installed next to the load cell, in order to blow any hot air away from it, and cool the steel shaft where it is connected to the load cell. These modifications minimized the heat effect on the accuracy of the load cell reading as shown in Fig. 7. Tests were conducted at

Table 2 Testing temperatures and the corresponding times needed to reach thermal equilibrium

Test's temperature, °C	Time to reach the desired test's temperature, min	Total time to reach thermal equilibrium, min
325	23	68
350	25	74
375	27	82
400	30	90
425	34	98
450	38	105
475	43	112
500	50	120

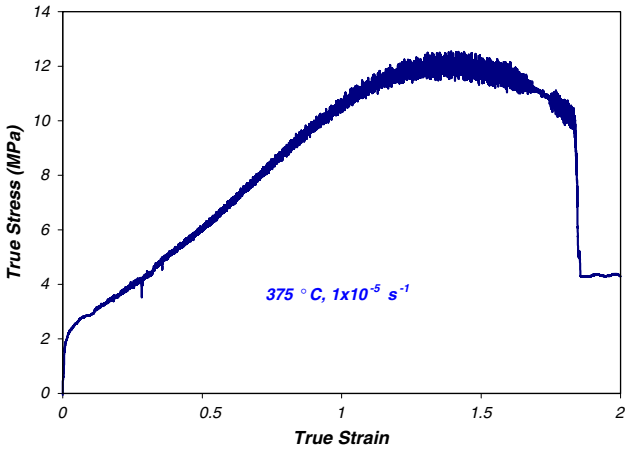

Fig. 6 Effect of heat on the load cell reading in a test at 375 °C and 10^{-5} s^{-1}

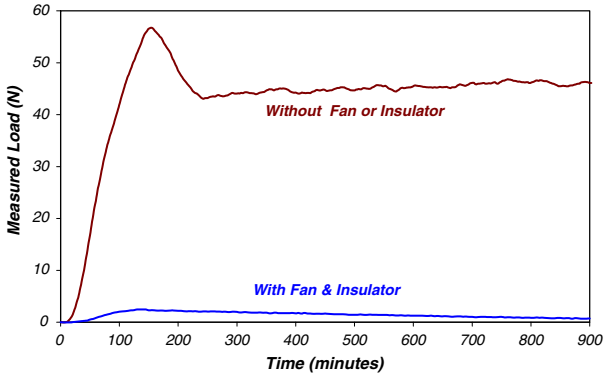

Fig. 7 Effect of heat on the load cell reading during zero-load tests at 500 °C

temperatures ranging between 325 and 500 °C and in all the tests, the reading of the load cell after fracture (complete separation) was always less than 2 N.

5. Constant Cross head Speed versus Constant Strain Rate Tests

The JIS H7501 sets the standard for testing superplastic materials by maintaining constant cross head speed (CCHS) during deformation (Ref 2) rather than constant true strain rate (CTSR). Superplastic materials are characterized by their flow stress sensitivity to strain rate, usually expressed by a sigmoidal-shaped logarithmic flow stress/strain rate curve. The sigmoidal curve should be constructed from a set of constant true-strain rate tests (CTSR) to accurately reflect strain rate sensitivity.

Figure 8 shows the results of two tests conducted at 375 °C and initial true strain rate of 5×10^{-4} s^{-1}. This strain rate value was kept constant during one of the two tests, while the corresponding cross head speed was maintained constant in the other test. The difference between the results is clear and becomes more significant as the deformation continues.

Additionally, in order to achieve maximum uniform deformation during SPF, the forming pressure cycle is usually designed based on a target strain rate selected from tensile tests.

Only constant true strain rate tests must be used in this regard for accurate description of deformation. This will become more important if an optimum loading path based on variable strain rates is used. Constant cross head speed tests will lead to underestimation of the desired strain rate and will shift the location of the desired strain rate jumps.

6. Evaluation of the Strain Rate Sensitivity Index *m*

In evaluating the strain rate sensitivity of the material, a common way to do it is by estimating the slope of the sigmoidal-shaped stress/strain rate curve at different points, representing different strain rates. The accuracy of this approach depends on the accuracy of the flow stress measurement in the first place. It has been shown earlier how remarkable the effect of some parameters, like heating time for instance, can be on the accuracy of the flow stress determination. For a more accurate evaluation of the index *m*, strain rate jump test is required. The two approaches are used here to determine the strain rate sensitivity of AZ31. Strain rate jump tests were conducted at 375 °C, covering strain rates from 2×10^{-5} to 2.5×10^{-2} s^{-1}. The upward jump was imposed at 0.2 strain, followed by the downward jump at 0.3 strain. Therefore, two estimates for *m* were made from each jump test;

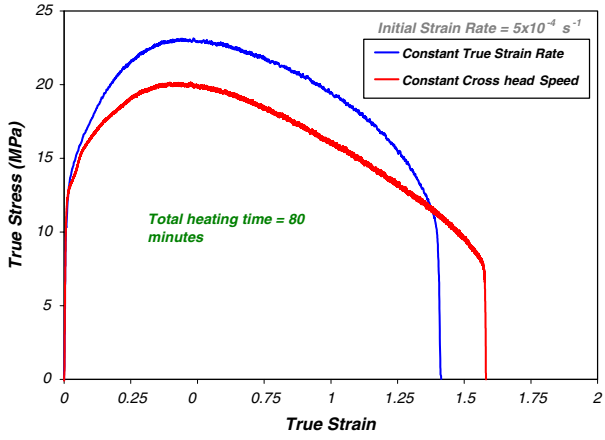

Fig. 8 Difference between a constant true strain rate and a constant cross head speed tests, at 375 °C and 5×10^{-4} s^{-1} initial strain rate

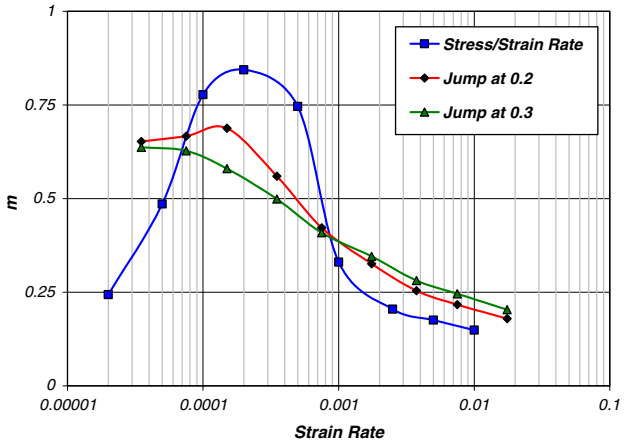

Fig. 9 Strain rate sensitivity index m at 375 °C; strain rate jump tests versus the stress/strain rate curve

m0.2 and m0.3, which are plotted in Fig. 9 as a function of strain rate.

Slight differences between the two estimates are observed, as expected, since the value of m depends on the strain (in addition to strain rate and temperature). Figure 9 also shows the m-value as determined from the slope of the stress-strain rate curve. Significant deviation from the strain rate jump test results is observed. Estimating m using the stress/strain rate curve reflects the strain rate sensitivity of the material at the very early stages of deformation (since the flow stress is determined at small strain values). While in a strain rate jump test, m is evaluated at some other strain value. In addition, evaluation of m using the stress/strain rate curve depends on the "smoothness" of the curve and erroneous results may be obtained.

Strain rate jump test is believed to be the more accurate way for evaluating m, although it is a common practice to estimate m from the stress/strain rate curves (Ref 2, 13, 14). The value of m is not only a qualitative tool used to examine whether a material exhibits superplasticity or not. Accurate m-value is vitally important for model development, calibration, and validation. Tests with multi-strain rate jumps are being carried out to establish the evolution of m with strain.

7. Conclusions

Although, the uniaxial tensile test is relatively the simplest testing procedure in characterizing the behavior of materials, however, once heat is involved, the simplicity turns into complexity. Superplasticity is achieved at high temperatures, making the involvement of heat unavoidable. The lack of a standardized testing procedure that can tackle the various issues associated with high temperature testing led to many contradictions regarding the behavior of superplastic materials and hindered researchers from compiling data from different sources to establish a reliable database for superplastic materials.

In this work, we have addressed some of the important issues associated with high temperature testing of superplastic materials in an attempt to highlight the need for developing standards for testing superplastic materials. Different tests have been conducted on the AZ31 Mg alloy to investigate the effects of various testing procedures and parameters on the accuracy of the results. Discussions and possible solutions have also been presented for a number of critical issues including gripping and test sample geometry, heating effects and required holding time and measurement of flow stress and strain rate sensitivity index.

It is hoped that this article will motivate discussions among researchers such that their experiences can be shared leading to a more accurate way of characterizing and testing superplastic materials.

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