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ON THE STANDARD TEST METHODS FOR EVALUATING THE TENSILE PROPERTIES OF METALLIC SUPERPLASTIC SHEETS

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Abstract

It is the extraordinarily large yet uniform tensile ductility that provoked a steady interest in superplastic materials in recent decades. This is clearly mirrored by the mounting research activities in the field, and the ever-growing number of components formed by the superplastic forming (SPF) technique. In spite of that, currently available standards on characterising the behaviour of this unique class of materials are very limited, and do not agree on many of the issues they cover. On the other hand, the standards fail to cover some of the important and controversial issues in superplastic materials' testing, and therefore leave the reader without any guidelines on those particular issues. In this work, a review of the three main standards that describe the proper method for testing superplastic materials (*JIS H7501*, *ASTM E2448* and *BS ISO 20032*) is carried out. The review focuses on the critical issues that significantly impact testing results, pointing out the points of agreement and disagreement among the three standards. And in an attempt to resolve some of these issues, specimen geometry, clamping device, heating and holding time; an integrated testing methodology (*test setup and testing procedure*) is presented. The methodology is centred on a recently-developed set of quick-mounting grips that facilitates mounting and retrieving the test specimen in a very short time. And coupled with the proposed specimen geometry and testing procedure, the methodology promises resolving the majority of the aforementioned testing issues. It is hoped that this work will provoke the development of a new universally-accepted standard method for testing superplastic materials at elevated temperatures.

Keywords:

Superplastic Metallic Sheets, Uniaxial Tensile Testing, Elevated Temperatures, Testing Standards, Quick-Mounting Grips.

1 INTRODUCTION

The uniaxial tensile test is probably the most common and the easiest testing procedure for characterising the mechanical behaviour of the various engineering materials. This is mirrored by the standardisation, or rather the versatility, of the test's elements (*specimen geometry, clamping device, test parameters*) to fit the different material classes; in addition to the widespread use of uniaxial load frames by industrial, academic and research facilities, where such standards are primarily adopted. It is, therefore, not surprising to deduce that the test of choice to study, characterise and model the unique class of superplastic materials is the uniaxial tensile test. Superplastic materials have been studied for several decades now, yet they seem to have gained a greater attention only recently. This is mainly because of the latest special interest in lightweight alloys (*titanium, aluminium and magnesium alloys*) for energy-saving potentials; and the superplastic forming technique is known to go hand-in-glove with these particular alloys!

In spite of the progress made on different fronts, and just like other research fields, there are several problems or limiting factors that hamper such progress on a larger scale. One of the unique problems in superplastic studies is the fact that data on superplastic materials is scattered and not reproducible among different parties; researchers investigating the same superplastic materials often report different (*even if slightly*) behaviours and properties. This

is quite expected, given that no standards on testing of these materials existed a decade ago! The first standard method for testing superplastic materials was published by the JIS, only in 2002 [1]; similar standards issued by the ASTM and ISO followed in 2005 and 2007, respectively [2,3]! On top of the lack of testing standards in recent years, the aforementioned standards have not been universally adopted in later testing activities, simply because they do not adequately nor accurately address some of the main controversial issues in testing of superplastic materials. It might be safe to say here that this is almost entirely driven by the condition at which these materials are tested; elevated temperatures. By referring to other standards that simply deal with tensile testing at higher-than-ambient temperatures, such as the ISO 783 [4] and the ASTM E21 [5], similarities in the drawbacks can be easily realised. One cannot find answers to several detrimental questions, particularly those related to the clamping (*gripping*) device, the heating path, holding time and testing procedure.

This work aims at resolving the major issues encountered in testing superplastic materials by going a step back to conventional elevated temperature tensile testing, and trying to tackle the problems introduced by the extreme temperatures. To do so, a review of the available related testing standards is first presented; in order to point out these issues, and draw conclusions about their interaction. Thereafter, a solution is proposed by means of an integrated testing methodology, hierarchically based on new quick-mount testing grips, followed by temperature-independent heating and holding times, and concluding with an accommodating testing procedure. Finally, and to prove its viability, the methodology is experimentally verified at selected testing conditions using AZ31 Mg alloy specimens.

2 REVIEW OF CURRENT TESTING STANDARDS

Three main standards, describing the proper method for testing superplastic materials, are currently available; JIS H7501 [1], ASTM E2448 [2] and BS ISO 20032 [3]. The ASTM standard is probably more detailed and descriptive than the JIS or ISO standards, which are quite similar in many aspects. Yet generally speaking, they all fail to provide any guidelines on specific testing issues (*heating/holding time*), and/or do not fully explain or provide a viable solution to others (*test specimen parameters and clamping device*). These will be discussed in detail here.

2.1 TEST SPECIMEN

While the JIS H7501 and BS ISO 20032 standards propose two specimen geometries, the ASTM E2448 proposes one. The flat parallel-sided specimens proposed by each of the three standards have different proportions in terms of their geometrical parameters, particularly in the fillet radius, the gauge length and the gauge/grip portion area ratio. None of the standards provide any guidelines on how those parameters were selected. On the other hand, the three standards do not agree on the definition of the gauge length; while it is the distance between the shoulders according to the ASTM E2448 [2], it is some selected distance within the parallel section according to the other two standards [1,3]. This difference has a significant impact on strain measurements, particularly with the hard-to-eliminate material flow into the gauge section.

2.2 CLAMPING (GRIPPING) DEVICE

This issue is the most influential of all, since it affects the selection of the specimen geometry, and has a direct significant impact on the most controversial issue of heating/holding time. The BS ISO 20032 standard hardly describes the clamping device.

The JIS H7501 inadequately covers the issue, and indicates that the gripping device should impose pressure on the specimen surface to provide the gripping action. This is highly questionable, since doing so would adversely affect material flow from the grip region during testing. The ASTM E2448 standard proposes and describes in detail a fairly simple clamping device; nonetheless, there are some drawbacks. For instance, they seem to be functional for testing specimens prepared from sheets with a specific thickness; it is not clear how the clamping device could accommodate other sheet thicknesses. Also, the keeper bar does not seem rigid enough to firmly hold the test specimen in place during the test, particularly for high strength alloys. More critical, the clamping mechanism is simple, yet not enough to offer the *optimum* means to grip the test specimen; that is, in the shortest possible time, with minimum effort, and with least impact on the heating/holding time (*will be further discussed later*). Unfortunately, other testing standards for elevated temperature uniaxial tensile testing, the ISO 783 [4] and the ASTM E21 [5], do not offer a better design for the clamping device.

2.3 HEATING TIME / HOLDING TIME

Undoubtedly, these two are the most controversial and least adequately discussed of all superplastic (*and even conventional elevated temperature*) tensile testing issues. The main divergence between the different standards starts with their different approaches for heating the specimen to the desired test temperature. The JIS and ISO standards [1,3,5] indicate that the specimen is heated (*alongside the testing setup*) from ambient to the desired test temperature, and so that is what dictates the heating time. While the ASTM standards [2,4] provide clear instructions for heating the furnace (*heating chamber*) to the desired test temperature before inserting the test specimen, and so heating time is simply the time needed for the furnace to re-establish the desired test temperature (*due to the interruption caused by opening the furnace, to insert and clamp the specimen*). The first approach (*JIS and ISO*) might be the easier one, particularly when testing at extremely high temperatures, where inserting the specimen into a hot furnace is troublesome. Yet this way of heating causes undesirable (*and mostly unpredictable*) changes to the microstructure of the material, which will be highly dependent on the test temperature; the higher the temperature, the longer the heating time, and consequently the greater those microstructural changes are going to be. Consequently, this will make standardising the issue of heating time (*and later holding time*) particularly difficult. The other approach, on the other hand, is the more logical way to heat the specimen, if the hardship of gripping the specimen is overcome and made consistent, regardless of the test temperature.

After reaching the desired test temperature, there is the issue of holding time; the time that needs to be allotted before imposing tensile strain on the specimen. This time should be long enough to assure thermal equilibrium in the test specimen, yet not too long to alter the microstructure of the material, and hence distort the superplastic properties being evaluated. Unfortunately, opinion is divided on the subject here too. Both the JIS H7501 and BS ISO 20032 standards leave the issue without any firm guidelines: “Interested parties shall agree on the time of heating the test piece to the test temperature and the holding time at the test temperature before starting the test, provided that such agreement shall be made with full assurance of uniform temperature distribution over the test piece” [1,3]. According to the ASTM E2448 and E21 standards, holding time is governed by reaching thermal equilibrium, which is detected by thermocouples readings, or when the cross-head beam ceases to move under the “protect specimen” control [2,5]; ASTM E21 dictates that this time should not be less than 20 minutes [5]. On the contrary, the ISO

783 stipulates a 10 minute holding time, regardless of the test temperature [4]. As depicted, these disagreements leave a lot of room for the reader to choose such a detrimental factor. In a previous work, it was shown that for the same material and test temperature, different heating and holding times have tremendous impacts on the resulting stress/strain curves [6].

3 PROPOSED TESTING METHODOLOGY

Motivated by the need to fill the gaps highlighted above, an integrated methodology for testing superplastic materials is sought. To address the majority of relevant questions, the interactions between the aforementioned major issues were analysed, and the following remarks were drafted as a starting point:

- Apart from specimen geometry, which could be partially isolated (*to some extent*), the remaining three issues are intimately related and highly dependent on each other.
- In order to make it viable, the test methodology has to tackle these three issues simultaneously, yet in a hierarchical order.
- The centre point of the methodology will be the clamping device, despite the fact that heating and holding times are more critical issues.

3.1 QUICK-MOUNT CLAMPING DEVICE (GRIPS)

Several considerations were taken into account, before designing a proper clamping device (grip):

- The design should be simple and functional, with minimal number of moving or sliding parts; this is to avoid potential complications associated with extreme temperatures.
- Tensile load should be exerted on (*and transmitted through*) the shoulders of the test specimen, and not the surface of the grip area.
- Pressure on the surface of the grip area should be avoided, in order to minimise material flow into the gauge area.
- The clamping device should be able to accommodate test specimens of different thicknesses.
- Most importantly, the design should allow for quick yet safe mounting and retrieval of the test specimen (*as will be demonstrated, this is a key feature in tackling the issues of heating and holding times*).

In view of this specification, the clamping device detailed in Figures 1-3 was designed and built. By referring to the detailed view in Figure 2, the device is merely composed of a grip body (1) and two L-shaped grip covers (2); the latter are fixed to the grip body's sides by two cap screws (3). A gap, just equal to the thickness of the specimen to be tested, is allowed for the specimen to slide into the grip. This gap can be easily altered to accommodate any thickness up to 0.25" (6.35 mm), since the grip covers are designed to glide along two slots on the sides of the grip body; this is clearly demonstrated by Figure 4. As depicted, the gripping action is simply achieved by sliding the specimen into the specified gap, which is aided by a slight chamfer on the inner edge of each of the grip covers. As a result, no pressure is applied to the grip area, hence material flow is minimised. Furthermore, the gripping action does not require sliding/moving/fastening any of the clamping device's few components, which is a key feature for the testing procedure presented below. More details about the clamping device can be found in [7].

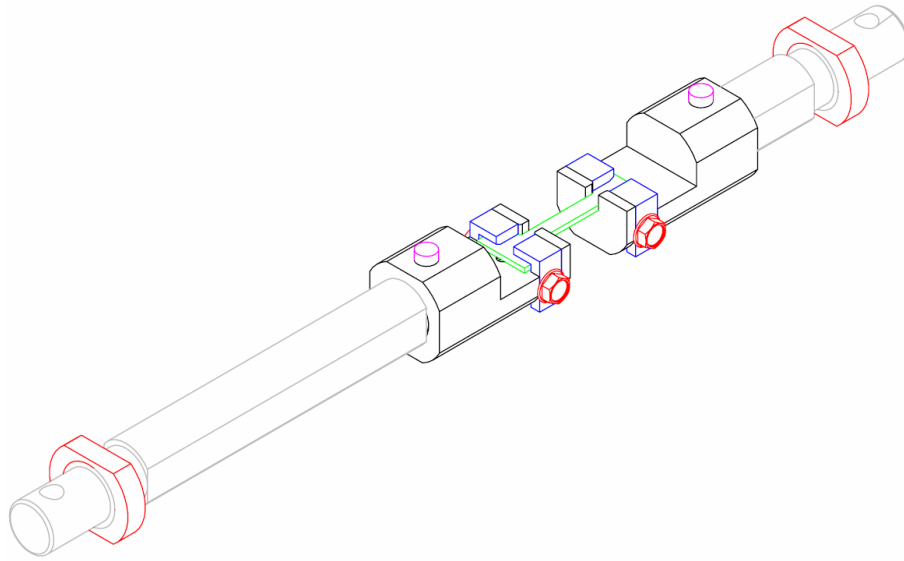


Figure 1: Overall isometric view of the clamping device

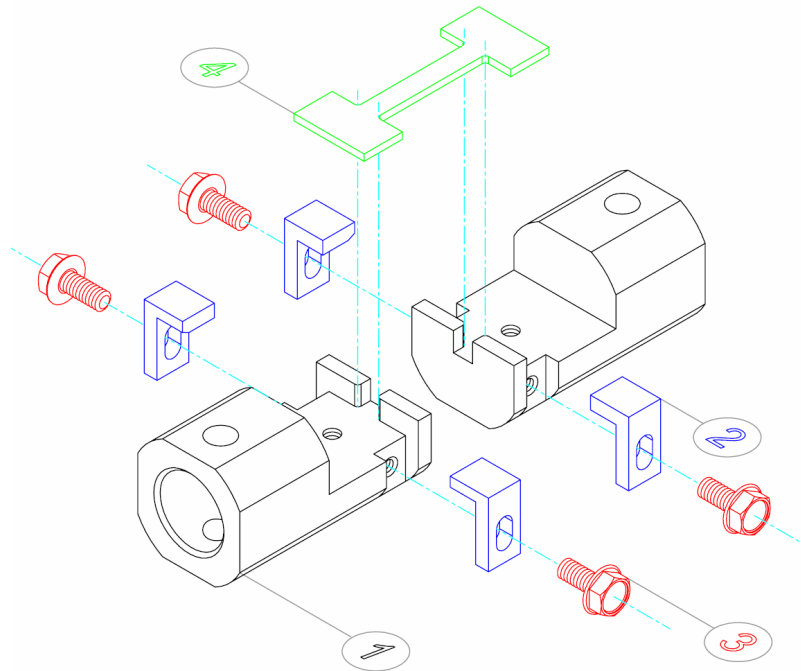


Figure 2: Detailed isometric view of the clamping device

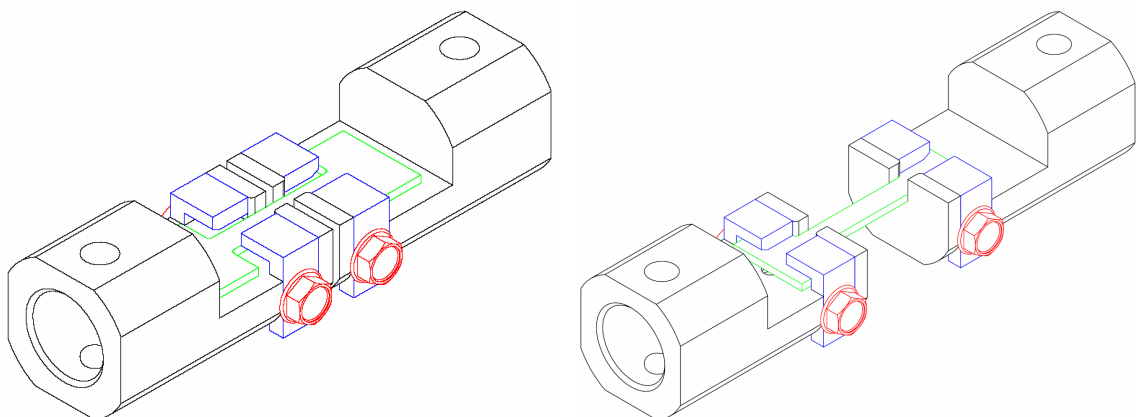


Figure 3: Isometric view demonstrating the gripping action (mechanism)

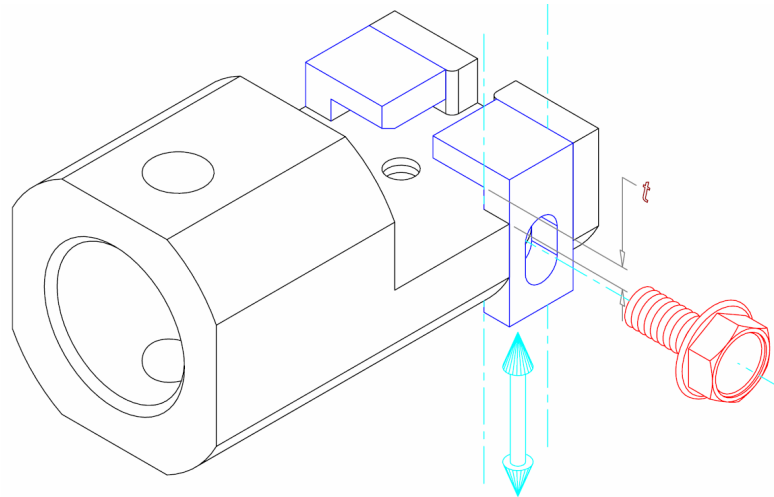


Figure 4: Detailed view demonstrating the ability to adjust the gap between the grip surface and grip covers to accommodate the varying thicknesses of test specimens

3.2 TESTING PROCEDURE

Initially, the test setup (*furnace and the clamping device within*) is heated to the desired temperature, and the cross-head beam is moved such that the distance between the two grips (*clamping devices*) is within a certain range, denoted by D_1 in Figure 5 (1). As shown in the figure, a spacing $\leq D_1$ guarantees the specimen to be inserted without any interference. A test specimen is then inserted into the space between the two grips as shown in (2), and left to slide by gravity into position (3). After that, the cross head beam is slowly moved upwards so that the top shoulder of the specimen becomes in contact with the upper grip (4), and until the bottom shoulder of the specimen is in contact with the lower grip too (5). The latter is sensed by monitoring the load cell reading, and making sure to stop as soon as the slightest force is detected. Finally, a certain holding time is allowed, and any needed adjustments (*to compensate for any minute expansion in the specimen*) are made, before the test is ready for initiation (6). It is important here to emphasise the specimen's unmatched freedom to expand thermally within the clamping device.

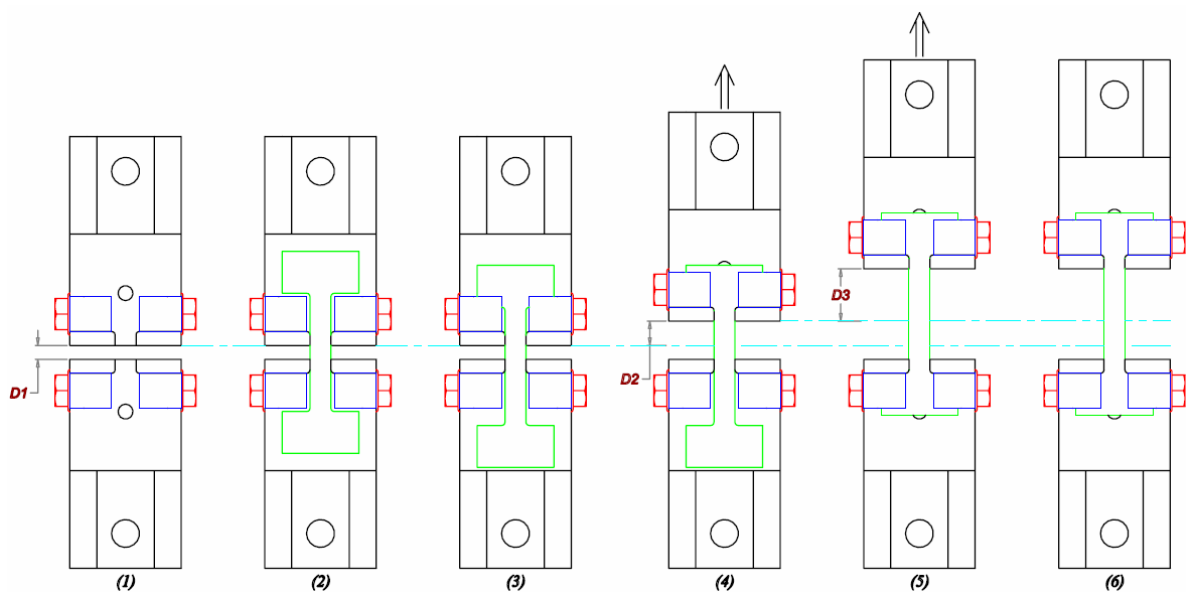


Figure 5: A schematic of the mounting/gripping mechanism

The described procedure, so far, should be done ideally without opening the furnace, and this is feasible from both the furnace and the clamping device perspective. Available furnaces or heating chambers often have an extra opening, excluding the top and bottom ones, through which the grip shafts extend. For instance, INSTRON's 3119 environmental chamber has the extra opening on top, while MTS's 651 chamber has it on the side. Considering the clamping device, on the other hand, one realises that its aforementioned features come into place here. There is the very simple mounting aspect, where there is no need to slide/move/fasten any parts. Recall that the operator's task is merely to deliver the specimen to the grips (*configuration (2) in Figure 5*); once in place, the remaining steps are taken care of remotely by moving the cross-head beam. In addition, no matter how crude the specimen delivery process might be, mounting simplicity guarantees minimal chance of exerting non-axial loads on the specimen, and hence causing a distortion. Moreover, note that the two clamping devices are not supposed to be spaced by an exact distance for the specimen to be located (*position (1) in Figure 5*). Conversely, if opening the furnace to insert the test specimen cannot be avoided, the clamping device still allows the mounting/gripping action in the shortest possible time. Therefore, two scenarios are considered next in setting the guidelines for determining the heating and holding times.

3.3 HEATING TIME / HOLDING TIME

Not having to open the furnace means near-elimination of the "heating time" issue, for two reasons. First; the furnace's temperature will be maintained almost constant, since the only possible disturbance is opening the insertion hole and introducing the specimen. Second; the test specimen is anticipated to reach the furnace's temperature soon after being inserted, due to its relatively small size, and temperature measurements can be made to determine the length of time precisely for the system being calibrated. Considering that situation, heating effects become less significant and independent of material and temperature, and the short time that actually goes into heating the specimen can be simply associated with "holding time". The latter can be easily determined for a specific system, and set to a small value (*no more than few minutes*) that implies the minimal changes to the microstructure of the material. If, on the other hand, the second scenario (*where the furnace had to be opened*) is followed, then both heating and holding times need to be determined through calibration and set. Nonetheless, it is more likely (*with the proposed clamping device*) to be able to standardise these two parameters, and assign them smaller values, for a given type of furnace, as will be shown next.

4 EXPERIMENTAL VERIFICATION

To demonstrate the viability of the proposed methodology, testing was carried out, as described above, following the two scenarios; by and without opening the furnace. The testing setup used here, and shown in Figure 6, consists of an 810 MTS servohydraulic load frame and a 651 environmental chamber. Test specimens were machined out of a 3.22mm (0.125") thick AZ31 magnesium alloy sheet, and tensile tests were conducted at 350 °C and initial strain rate of 0.0004 s⁻¹. This material was picked for its good superplastic behaviour at a wide range of temperatures and strain rates. To ease monitoring both deformation and material flow within the specimen, a circular grid pattern was imposed on the surface of each test specimen by electrochemical etching, as shown in Figure 7.

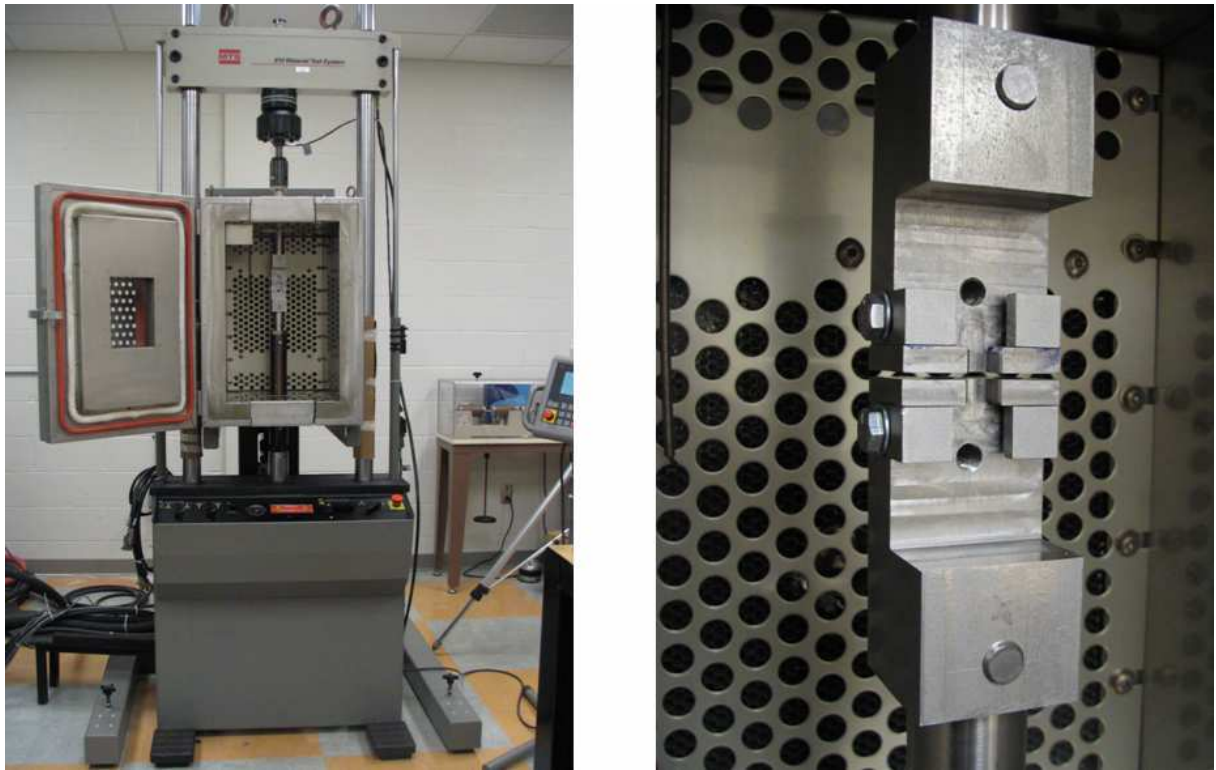


Figure 6: Testing setup and the clamping device

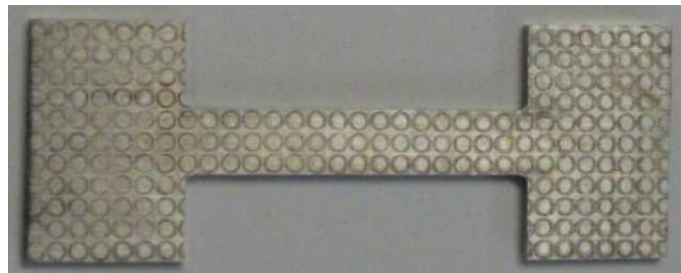


Figure 7: A circular grid pattern imposed on the test specimen

4.1 WITHOUT OPENING THE FURNACE

The setup was first heated to the test temperature, and then left to equilibrate for 15 minutes. The top hole was partially opened, and a test specimen was slowly inserted, and then gently pushed in place (*the gap between the two grips*). The process of mounting the specimen was trouble-free, and took about 30 seconds. During the process, no significant drop in the chamber's temperature was noticed. With the specimen in place, a total of three minutes (*including the 30 seconds of the mounting procedure*) was arbitrarily allowed before straining the specimen. As previously discussed, this heating/holding time interval can still be agreed on by the different interested parties; yet with the proposed clamping device and testing methodology, it is significantly shorter and therefore unproblematic to standardise.

4.2 BY OPENING THE FURNACE

After heating and equilibrating again to the test temperature, another specimen was loaded into the grips by opening the furnace's door. Similarly, the process was found to be trouble-free, and took only 5 seconds from start to end. Expectedly, the temperature dropped during these few seconds; nonetheless, the drop was no greater than five degrees

centigrade, and the test temperature of 350 °C was re-established after less than 45 seconds. Similarly, with the specimen in place, three minutes were allowed before beginning the test.

The tested specimens were stretched to 300% elongation, and Figure 8 below shows a close-up of the material deformation within a selected specimen. Given that material flow is inevitable, nevertheless, it is still acceptable in this case. Naturally, we would expect more flow at higher temperatures and strain limits, yet what is more important is the ratio between material flow and the overall stretching in the specimen. And with the small amount of material flow in Figure 8, it is safe to deduce that this will not be an issue at more severe testing conditions.

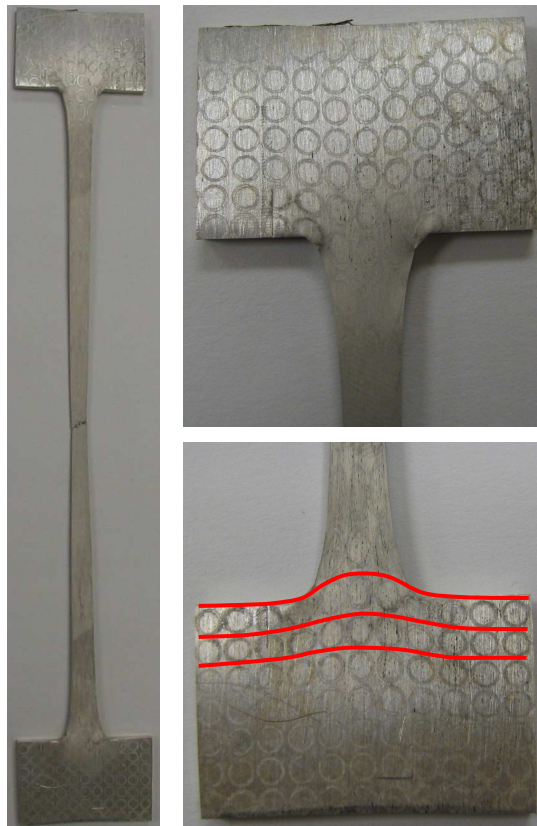


Figure 8: A circular grid pattern imposed on the test specimen

Finally, the limitations of the available testing setup did not permit the investigation of the material flow issue at higher temperatures and strain limits; nevertheless, it does not reduce the great potential (*shown by the preliminary results*) of both the proposed clamping device and the testing methodology, in resolving the more critical issues in elevated temperature superplastic testing.

5 SUMMARY

This work presents an effort to resolve the critical issues encountered in uniaxial tensile testing of superplastic alloy sheets at elevated temperatures. A review of available standards indicates that the three main standards on the subject fail to provide enough guidelines, at the anticipated level of detail. Therefore, quick-mount grips were designed and used here as the centre point of an integrated testing methodology, which describes the grips' ability to facilitate mounting the specimen in a very short time, thereby, helping to resolve the critical

issues of heating and holding times. Tensile tests at selected conditions were carried out to test the practicality of the grips and the viability of the methodology, and they were shown to be promising from more than one perspective. Conclusively, the proposed methodology offers more answers than questions, and lays the ground for tackling the critical testing issues; yet it leaves enough flexibility with respect to minor aspects and details, for the interested parties to agree on. This effort should not be considered a proposal for a new testing standard, but rather an initiation for a much needed development of a new universally-accepted standard method for characterising the behaviour of superplastic materials.

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