Development of a Diffusion Bonding Route for Joining Oxide-Dispersion-Strengthened (ODS) Steels for Nuclear Applications

JOSÉ RODOLPHO DE OLIVEIRA LEO and MICHAEL E. FITZPATRICK

Oxide-dispersion-strengthened (ODS) steels are candidate materials for components in current and future nuclear power plants. One of the issues with using ODS steels is the difficulty of joining them without loss of mechanical performance. In this study, austenitic ODS 316L stainless steel was diffusion-bonded to Inconel 718 superalloy. Having optimized the bonding conditions, a number of samples were made at 1200 °C with a bonding time of 1 hour and pressure of 10 MPa. Preliminary mechanical and microstructural analyses indicated the formation of a sound joint interface, despite slight grain growth in the ODS 316L. A post-bonding thermo-mechanical treatment (TMT) was conducted to refine and restore the microstructure of the ODS 316L. Comparative TEM investigations of the parent ODS alloy and the bonded samples (with and without TMT) along with statistical analyses showed that the Y–Ti–O oxide size distribution remains unaffected by the bonding and complementary TMT cycles, indicating stability of such particles even at very high temperatures and suitability of the devised route for joining the ODS 316L steel.

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I. INTRODUCTION

ENGINEERING application of oxide-dispersion-strengthened (ODS) steels is limited due to the difficulty in joining them to themselves and to other materials. Conventional welding techniques cause segregation and clustering of the oxide particles, which can lead to joint embrittlement. Solid-state diffusion bonding is regarded as a promising joining technique for ODS steels, since it is carried out below the melting point of the alloy, therefore averting redistribution or clustering of the oxide particles.1,2

Austenitic stainless steel SS316L is a promising matrix for ODS materials due to its high corrosion resistance, desirable mechanical properties, high-temperature strength, toughness and creep resistance.3-6 The SS316L ODS alloy is of interest in power plant applications, particularly in nuclear fission reactors, mainly due to their superior resistance to irradiation damage.7

Diffusion bonding is a solid-state joining technique in which two or more parts are kept in contact under a constant pressure and at high temperatures, normally above 80 pct of the absolute melting point of the parts, for time intervals ranging from a few minutes up to several hours.2 This process has been identified as a potential choice for joining parts made of ODS alloys,1,8-11 since it relies on interatomic diffusion across the joint interface, rather than the formation of a molten pool associated with conventional fusion welding processes. A suitable combination of pressure, temperature, and time, using an interlayer in some cases, is required in order to achieve high-strength bonds. For instance, the bonding temperature should be high enough to create a defect-free interface and, at the same time, not too high to alter the microstructure and properties of the original materials.

A particular aspect of diffusion bonding that has been gaining increasing popularity is the production of dissimilar metal joints. Specifically, dissimilar joints between austenitic stainless steels and nickel superalloys are of interest in the production of components to be used at high temperatures, finding application in the aerospace and nuclear energy sectors.12 The joining of 316L stainless steel to Inconel 718 via solid-state diffusion bonding has been the focus of a few works. Locci et al. have investigated the production of joints between these materials with and without an
interlayer,\textsuperscript{[13]} while Li et al., investigated the use of electrically assisted solid-state joining for producing joints between the same alloys.\textsuperscript{[12]} The present study is the first to join austenitic ODS 316L stainless steel to nickel-based superalloy Inconel 718 via diffusion bonding, motivated by the requirement potential structural materials for advanced nuclear power plants are expected to fulfill of being joined to different alloys. The optimum bonding conditions were determined, and the effects of the bonding cycle, as well as of a post-bonding thermo-mechanical process, on the microstructure and mechanical behavior of the bonded samples were investigated. The joints were assessed by tensile and creep tests, along with optical microscopy and TEM characterization of the resultant microstructural features.

II. EXPERIMENTAL PROCEDURE

A. Initial Bonding Trials with Inconel 718 and ODS 316L Bonded Discs

The austenitic ODS 316L stainless steel used in this investigation is an alloy with 0.35 pct wt yttria, fully characterized elsewhere.\textsuperscript{[14]} Inconel 718, a nickel-based superalloy, was chosen to be diffusion bonded to the ODS 316L due to its high strength, especially at high temperatures. With this, it was expected that the mechanical behavior of the bonded samples would be as similar as possible to the parent ODS 316L. The chemical compositions of both materials are given in Table I.

Disc-shaped samples with 8 mm diameter and 5 mm thickness were wire EDM cut from the parent ODS 316L and Inconel 718 alloys, and prepared for bonding by having their cross sections ground with emery paper to fully remove debris and the ~10 μm porous layer left from the cutting process, a procedure that improves the quality of the bond interface, in terms of cleanliness and mechanical properties.\textsuperscript{[2]} Samples were then bonded in vacuum using a purpose-built diffusion bonding rig. Diffusion bonding trials were performed at different temperatures, ranging from 1100 °C to 1250 °C, and bonding times up to 1 hour. A 200 kgf load was initially applied, and then reduced to 50 kgf during the process to avoid excessive deformation of the sample. Preliminary manual bending tests indicated that bonding below 1200 °C produced weak joints irrespective of the bonding time, since samples failed either at the joint under minimal applied bending force or during the cutting of slices in preparation for the test. On the other hand, bonds made at 1250 °C promoted undesirable alterations in the microstructure due to excessive diffusion of nickel into the ODS 316L matrix. It was ascertained that bonding at 1200 °C for 1 hour with a starting pressure of about 10 MPa represented the best parameters.

Thin slices (less than 1-mm thick) were cut from the bonded sample, as schematically shown in Figure 1(a), and used for optical microscopy and for primary assessment of the quality of the bond by means of the same manual bending tests. These latter consisted of bending the thin slices across their bond lines until they cracked and broke from the bonding interface or withstood at least 90° bending angle without joint failure.\textsuperscript{[15]} Samples for optical microscopy were mounted and ground using 1200 grit emery papers, polished with a 1 μm diamond particle suspension, and etched in 60 pct nitric acid. The microstructures of the as-received and bonded samples were characterized by a Leica DMI 5000 M optical microscope, and the grain size was measured according to ASTM E112.

B. Diffusion Bonding of Specimens for Mechanical Testing

With the bond strength and process conditions optimized, diffusion bonding was applied to the fabrication of hybrid specimens. For this purpose, instead of discs, a “sandwich” sample was devised, consisting of two Inconel 718 cylinders of 13 mm diameter and 35 mm length, and, in between them, a disc of ODS 316L with the same diameter and 5 mm thickness, as shown in Figure 1(b). The parts to be diffusion-bonded were cut by wire EDM, and their preparation for the joining process involved the same steps previously described, except that, due to the length of the Inconel 718 cylinders, their lateral surfaces were also ground with abrasive paper to remove the EDM cutting layer. While this latter does not necessarily impact material properties, best cleaning practices are followed, in order to avoid having any surface debris evaporating and accumulating in the turbopump of the machine, due to the high temperatures and high levels of vacuum, potentially causing damage.\textsuperscript{[1]}

Following preparation, the parts to be diffusion bonded were properly aligned and assembled onto the machine load train. A type-K thermocouple was spot-welded to the sample. An induction heating coil with four spirals of 60 mm diameter covering, uniformly, approximately 60 pct of the central portion of the aligned parts was wrapped around the specimen. The choice of the coil is crucial for the success of the bonding. An inadequate size of coil means that the spirals will not be equally distributed along the sample, so that non-uniform heating occurs.

Heating and cooling rates were fixed at 200 °C/min. Similarly to the trials, a load of 200 kgf was applied during the first 5 minutes of the process and, then, reduced, in steps of 50 kgf every 5 minutes, to a value of 50 kgf, which was maintained during the remaining bonding time. Maintaining the initially applied load results in additional stress on top of the stresses induced by the impeded thermal expansion, which could plastically deform the materials and change the geometry of the samples.

From this batch of diffusion-bonded samples, specimens for mechanical testing were machined. Room temperature tensile specimens were designed following BS EN 10002-5,\textsuperscript{[16]} modified for testing in a mini-tensile test rig. Creep specimens were prepared from a modified design of the same standard. Further details on the tests are provided in the following sections.
C. Room Temperature Tensile Test of the Hybrid Specimens

Mini-tensile test specimens with 12 mm gauge length, overall length of 44 mm and a gauge section of 3 mm by 1 mm were designed in accordance with BS EN 10002-5 1992 and cut from the bonded cylinders. In order to conduct the tests, an MTI/Fullam tester machine was used with maximum load capacity of 4.5 kN and fitted with two moveable crossheads driven by a worm gear system actuated by a direct current motor. A displacement rate of 0.096 mm min\(^{-1}\) was used for the tensile straining, whose purpose was to take the sample to rupture and evaluate the location where it occurred. A strain gauge was attached to the central portion of the specimen, corresponding to the ODS 316L segment.

D. Hybrid Specimens for Creep-Rupture Testing

A hybrid creep-rupture test sample was also produced with dimensions scaled from BS EN 10002-5; however, it had to be adapted to fit a special creep test rig. The specimen was also machined with 12 mm gauge length and overall length of 44 mm, and 1.5 mm by 1.5 mm gauge cross-section. Creep-rupture testing was carried out using an appropriate apparatus for high-temperature creep tests in miniaturized specimens. A thermocouple was spot-welded to the ODS 316L part of the gauge length. The specimen was crept under 200 MPa at 650 °C, with this temperature maintained at ±1 °C. Optical microscopy was used for determining the grain sizes of both materials and establishing a comparison with the as-received condition. Finally, a fractographic study was carried out in a Zeiss Supra 55VP Field-Emission-Gun SEM operated at 5 keV.

E. Post-bonding Thermo-mechanical Treatment (TMT) of the Samples

A plan was devised to promote refinement of the microstructure and achieve a condition of homogenized grains, particularly for the ODS 316L, subsequent to the diffusion bonding of the materials. The plan consisted of introducing some deformation to the microstructure, in order to create sites where nucleation of new grains could take place during the subsequent heat treatment step. This latter was carried out by heating the diffusion-bonded materials to the estimated recrystallization temperature of the ODS 316L, keeping them at this level for a determined time and cooling them outside the furnace (air-cooling), in order to prevent significant diffusion, thus avoiding grain growth.

### Table I. Chemical Composition of the Alloys Used in This Work

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Ni (+Co)</th>
<th>Cr</th>
<th>Fe</th>
<th>Nb (+Ta)</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
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<tr>
<td>Inconel 718</td>
<td>55</td>
<td>17</td>
<td>bal.</td>
<td>5.5</td>
<td>3</td>
<td>0.9</td>
<td>0.5</td>
<td>0.08 max</td>
<td>0.35 max</td>
<td>0.35 max</td>
</tr>
<tr>
<td>ODS 316L</td>
<td>13.23</td>
<td>16.82</td>
<td>bal.</td>
<td>—</td>
<td>2.48</td>
<td>0.28</td>
<td>0.30</td>
<td>0.008</td>
<td>0.72</td>
<td>0.40</td>
</tr>
</tbody>
</table>

Fig. 1—Schematic of the bonded discs and the longitudinal cut for extracting the slices (a), and a picture of the “sandwiched” sample mounted for bonding (b).
Slices of approximately 0.75 mm thickness were longitudinally cut from the diffusion-bonded discs, similarly to the schematic of Figure 1(a), and subjected to a thermo-mechanical treatment comprising cold-rolling as the work-hardening stage and an annealing treatment carried out at 1150 °C for 0.5 hour, followed by air-cooling. The parameters for the heat treatment were chosen based on the high temperatures associated with the recrystallization of ODS steels, [17,18] and on the values used by Wang et al. [19] on an ODS 310 austenitic steel. The work-hardening stage was set to produce deformations of 5, 13 and 25 pct on the slices. These percentages were based on the works of Abou Zahra and Schroeder, [20] and Engelberg et al., [21] in an attempt to achieve the lowest imposed deformation possible that was still higher than the critical level for recrystallization. Before the cold rolling was applied, the initial thickness of the slice was measured with an electronic micrometer with 0 to 25 mm measuring range and ±0.001 mm precision. A manual cold-rolling device was used to reduce the thickness of the slices according to the aforementioned levels of deformation.

The normalization annealing was then carried out in a vacuum furnace. An N-type thermocouple ensured the temperature remained at 1150 ± 1 °C. The slices were then removed from the furnace and left to cool in ambient air. Each slice, including the as-diffusion-bonded used as control, was ground, polished, and etched (as described in Section II–A) for investigations on the optical microscope.

F. TEM Examination of the Oxides Post-TMT

To evaluate whether changes may also have occurred for the particle size distribution, TEM studies were carried out for the as-diffusion-bonded slice (not TMT-treated) and for the slice subjected to 25 pet deformation plus heat treatment, in order to check for possible differences in comparison to the oxide size distribution of the as-received ODS 316L. [14] TEM foils were extracted from the slices, ground with SiC papers of 40, 15 and 5 µm and cut to 3-mm-diameter disks. These were further thinned by jet electropolishing in a 5 pet perchloric acid and 95 pet ethanol solution at −50 °C. The foils were examined using a JEOL JEM 2100 Transmission Electron Microscope operated at 200 kV. A total of 278 particles were measured for the TMT ODS 316L, counted in three foils, from 15 images; while 301 particles were checked in the as-diffusion-bonded material, in 19 different micrographs taken also from three foils. A statistical analysis based on hypothesis tests was conducted, then, for the as-received, the as-bonded and bonded-plus-TMT oxide size distributions, to set appropriate comparisons.

III. RESULTS

A. Microstructural Examination

Optical microscopy showed the presence of equiaxed grains with a size of 106 ± 12 µm, in the as-received Inconel 718 alloy; and elongated grains, with an average grain size of 25 ± 3 µm in the as-received ODS 316L, as seen in Figure 2.

Figures 3(a) and (b) show that the samples bonded at 1200 °C for 1 hour have a straight bond line without any detectable defects such as voids or unbonded areas. There is little or no change in the microstructures of both ODS 316L and Inconel 718 due to exposure to high temperature.

B. Room Temperature Bending and Tensile Tests

Thin slices cut from the bonded discs were subjected to bending tests. Figure 4 shows one of the slices bent into a U-shape, with a bending radius of 2 mm, without any failure in the joint. It can be noted that the severe plastic deformation experienced by the steel resulted in the “orange peel effect”, whereas there is hardly any change on the surface the Inconel. Also, no signs of fracture are observed, and there is no visual indication of the location of the bond line.

Figure 5(a) depicts a room temperature tensile test curve for the hybrid, “sandwiched” bonded sample shown in Figure 5(b). Details of the specimen design are presented as an insert in Figure 5(a). The stresses were calculated from the readings of load, which were then divided by the rectangular cross-sectional area of the sample. All specimens failed within the Inconel 718, at
an ultimate tensile strength of around 500 MPa. The bond interface and the ODS 316L segment of the specimens remained intact. It is important to mention that Figure 5 does not show the plot of the whole dataset from the tensile tests, due to limitations in the data recording system of the testing device, and that strains were recorded within the ODS segment of the test sample only. Post-test examination of the samples suggested overall ductility of 3.5 to 4 pct.

C. Creep-Rupture Test

The creep-rupture test, conducted on the specimen shown with its design dimensions in Figure 6(a), provided additional evidence of a good integrity bond, since the failure of the specimen crept at 650 °C under 200 MPa occurred within the ODS 316L segment in the gauge length, but far from the bond lines, as illustrated in Figure 6(b). As seen from the creep curve shown in Figure 7, rupture occurred after just over 72 hours, with a corresponding creep ductility of almost 11 pct total strain.

Optical microscopy surveys for the materials in the crept specimen showed pronounced growth of the Inconel 718 grains, in comparison to the as-received condition, but no changes (within the measurement accuracy) for the ODS 316L. The average grain sizes from five different areas of survey for the ODS 316L and the Inconel 718 after the bonding cycle and creep deformation are summarized in Table II, along with the reported sizes for the as-received materials. Another interesting feature is found in the Inconel 718 part, with what seems to be δ-phase precipitates inside the grains and along grain boundaries, as seen in Figure 8. The bonding cycle takes the material to a level above the solvus temperature, so that, upon cooling, precipitation of this orthorhombic Ni3Nb phase is expected to occur.[22]

The SEM fractographic examination of the ruptured surface of the crept specimen is presented in Figure 9, which suggests that the failure occurred via a mixed mode of crack propagation: most areas presented considerable ductility, but some failed in a brittle manner. Figure 9(a), a low-magnification general aspect of the rupture surface, reveals extensive dimpled regions with cracks. When magnified, some portions of the topography showed the presence of cleavage, as depicted by Figure 9(b), where the smooth surfaces with some cracks indicate the occurrence of brittle fracture through them. Most portions of the specimen, on the other hand, when observed under higher magnification, showed preserved grains surrounded by dimples (Figure 9(c)), suggesting that the crack propagation in these regions happened in a ductile manner, with both inter- and intra-granular failures being preceded by significant deformation.

D. Thermo-mechanical Treatment (TMT) Results

Table III lists the deformation levels imposed on each cold-rolled slice, according to the intended deformation level. The initial and measured final thicknesses represent the average of three measurements for each slice. Slices 1 and 3 presented a uniform thickness along their extensions, but slice 2 showed significant non-uniformity, with variations in its thickness. As seen from Table III, the imposed deformation during cold rolling matched the intended level in all cases.

The results from the grain size measurement applied to the slices after the complete thermo-mechanical treatment are summarized in Table IV. The as-diffusion-bonded slice was not subject to the TMT, hence the
agreement between the average grain sizes found here with those from the characterization of the bonded materials. The optical micrographs of the ODS 316L after the TMT for each plastic strain level are presented in Figure 10. As can be seen, only a plastic strain level of 25 pct produced refinement of the steel microstructure.

Fig. 5—(a) Plot of tensile curve of the diffusion-bonded sample, whose dimensions are presented in the insert; (b) a picture of an in-test tensile specimen, with the strain gauge attached to the ODS 316L steel part; (c) A tested specimen moments after its failure, showing the relative position of the rupture line with regards to the joints. Note that the plot does not show the entire data to failure. The UTS was around 500 MPa, and overall sample ductility estimated at 3.5 to 4 pct.

Fig. 6—(a) Hybrid creep-rupture specimen details; (b) the crept specimen in the fixtures, showing the failure well inside the ODS 316L steel segment.

Fig. 7—Creep-rupture test curve for the hybrid specimen tested at 650°C and 200 MPa.

E. Effect of the Thermal Cycle on the Oxide Particles

Given that the temperatures envisaged for the diffusion bonding process and the recrystallization heat treatment are high enough to cause increased diffusion rates, the distribution of oxide particle sizes were measured, using contrast-based image processing techniques, for the as-bonded and for the slice subjected to 25 pct plastic strain, given that this level of deformation
was the only one to trigger recrystallization. The size distributions are shown in Figures 11 and 12, and are very similar.

IV. DISCUSSION

A. General Analysis of the Properties of the Bonded ODS 316L

The development of good joint efficiency, with appropriate service performance, is dependent on several factors, such as surface cleanliness, surface roughness, applied pressure, vacuum level in the chamber and, most importantly, the bonding temperature.\[23]\] This latter determines to what extent the stages of metal-to-metal contact through plastic deformation, the preliminary diffusion across the interface with recrystallization, and the volume diffusion towards the voids at the interface happen. Given that, in all tests carried out, the parameters deployed were the same, except for the temperature, it is reasonable to infer that, in the bonding trials below 1200 °C, these stages have not been sufficiently developed, and hence require longer periods of time to produce sound bonds than the ones deployed (of up to 1 hour).\[13]\] In particular, the last stage, related to bulk diffusion towards the remaining voids in the joint, as well as grain boundary movement,\[24]\] seems to have been restricted, since the diffusion coefficients of the diffusing species have an exponential relationship with the temperature.\[23,25]\] This explains the poor performance of the resulting joints in these trials.

The bonding conditions used for joining ODS 316L to Inconel 718 after the trials produced a sound and clean joint. The interfaces created by bonding at 1200 °C for 1 hour were smooth, preserved the microstructure of the materials, and exhibited good mechanical behavior in both room temperature tensile testing and in creep testing at 650 °C and 200 MPa. In the case of the tensile tests, the failure occurred in the Inconel 718 region, suggesting the nickel acted as the limiting material, at room temperature, for the joint. The ductility reported in the present study falls below the commonly reported baseline of, at least, 20 pct straining at failure for Inconel 718,\[26,27]\] and around 40 pct for the ODS steel.\[14]\] Such low ductility levels at room temperature recorded during tensile testing are comparable to those observed at 650°C in stress rupture tests.\[28]\] It is important to mention that, because the strain gauge was attached to the central portion of the specimen, on the ODS 316L segment, the readings obtained do not reflect the deformation individually experienced by each material the bonded sample. It is possible that strains were concentrated in the parts of the specimen gauge length made of Inconel 718, away from the segment to which the strain gauge was attached.

The creep test showed that the joint between the ODS 316L and the Inconel 718 preserved its integrity and demonstrated good strength also at high temperature. The fractography of the crept specimen is consistent with the observed creep ductility, considering the shallow aspect of the few brittle cracks. Also, the absence of torn grain surfaces indicates that most of these cracks propagated in an intergranular way, while the extensively dimpled surface is a consequence of a predominantly ductile failure, as seen in Figure 9, being a reasonable result for a material that exhibited almost 11 pct of creep ductility.

All these investigations pointed to the necessity of a post-diffusion bonding theromechanical treatment (TMT), for retrieving, or improving, the performance of the joined materials, in particular the ODS 316L. Results showed that deformation levels of 5 and 13 pct

<table>
<thead>
<tr>
<th>Material</th>
<th>Grain Size Before Bonding (µm)</th>
<th>Grain Size After Diffusion Bonding &amp; Before Creep (µm)</th>
<th>Grain Size After Diffusion bonding &amp; Creep (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ODS 316L</td>
<td>25 ± 3</td>
<td>26 ± 3</td>
<td>29 ± 3</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>106 ± 12</td>
<td>378 ± 12</td>
<td>422 ± 13</td>
</tr>
</tbody>
</table>

Table II. Average Grain Sizes Before and After Diffusion Bonding

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Fig. 8—Optical micrograph of the bond line of the failed crept specimen.
did not trigger enough recrystallization to cause significant change in the grain size of the ODS 316L. The slice deformed to 25 pct in cold rolling, on the other hand, presented a very satisfactory result, showing refinement of the microstructure almost completely. Recrystallized grains were homogeneously distributed throughout the ODS 316L structure. From the various conditions tested, this level of deformation was the only one above the critical plastic strain for recrystallization.

Although not being the main concern in this study, the changes in the Inconel 718 are worth an appraisal. In most of the cases, its grains experienced just a slight growth. This can probably be attributed to a less pronounced work-hardening of the nickel superalloy when cold-rolled, due to its superior strength, which may have induced fewer defects and, thus, provided fewer sites for recrystallization. As a consequence, only a slight grain growth was observed. The exception was slice 3, in which larger grains were observed. As no clear reason could be discerned for such a change, it is likely that the observed grain size was already present in the Inconel 718 batch, as a variation of the material, before any treatment was done.

B. Effects of the Thermal Cycle on the Oxide Particles

Although the TMT previously described showed that a certain combination of parameters was effective in refining the microstructure, the material of interest is an ODS steel, for which, equally important to the grain refinement, are the possible effects that the exposure to high-temperature thermomechanical processing might have on the nanometric oxide particles. These oxides are known for being insoluble at high temperatures, even close to the melting point of the steel, and very stable, strongly resisting coarsening at temperatures in excess of 1000 °C. The present work confirmed this feature of the nanometric oxides of the ODS 316L, as seen in Figure 13, which provides a comparison of the size distribution between the as-diffusion-bonded and the TMT-treated materials.

Even though the temperatures envisaged for the diffusion bonding process and the recrystallization heat treatment are high enough to cause elevated diffusion rates, the size distributions were practically the same, as observed from Figures 11, 12 and 13. No significant statistical difference was found between the as diffusion-bonded sample and the TMT treated one, as shown in Table V for a hypothesis test for the size distribution in these two conditions; and no evidence of growth of the oxides after the thermal cycle due to the bonding and to the TMT was found, in comparison to the as-received ODS 316L. A hypothesis test carried out for the as-received and diffusion-bonded and TMT-treated steels indicated equivalence between both oxide size distributions, as summarized by Table VI. These results are consistent with the findings from several studies that reported stability of the oxide nanoparticles for ODS steels annealed at 1200 °C for up to 24 hours.

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Fig. 9—(a) Low-magnification SEM fractography showing the dimpled areas with cracks. The areas highlighted by the red and yellow circles are magnified in (b) cracked smooth surface, and (c) dimples around grains, respectively (Color figure online).
It must be emphasized, though, that, in all these investigations (including the present study), the oxide particles were of Y–Ti–O or Y–O composition. Therefore, plans for a successful TMT need to take into consideration the nature of the oxides, as different compositions will react differently to exposure to a very high temperature and/or to a long period of time, and the influence of these parameters must be continuously investigated.

Table III. Conditions for Cold Rolling of the Slices Cut from the Diffusion-Bonded ODS 316L/Inconel 718 Discs

<table>
<thead>
<tr>
<th>Slice</th>
<th>Initial Thickness (mm)</th>
<th>Measured Final Thickness (mm)</th>
<th>Imposed Deformation (Pct)</th>
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<tbody>
<tr>
<td>Slice 1</td>
<td>0.664</td>
<td>0.628</td>
<td>5.4</td>
</tr>
<tr>
<td>Slice 2</td>
<td>0.786</td>
<td>0.678</td>
<td>13.7</td>
</tr>
<tr>
<td>Slice 3</td>
<td>0.782</td>
<td>0.582</td>
<td>25.6</td>
</tr>
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</table>

Table IV. Grain Size Measurement for Each Slice

<table>
<thead>
<tr>
<th>Materia/Slice</th>
<th>As diffusion-bonded</th>
<th>Slice 1 (5 Pct)</th>
<th>Slice 2 (13 Pct)</th>
<th>Slice 3 (25 Pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ODS 316L</td>
<td>26 ± 3</td>
<td>26 ± 5</td>
<td>27 ± 7</td>
<td>19 ± 2</td>
</tr>
<tr>
<td>Inconel 718</td>
<td>378 ± 12</td>
<td>452 ± 42</td>
<td>439 ± 17</td>
<td>609 ± 69</td>
</tr>
</tbody>
</table>

All measurements are in μm.

Fig. 10—Optical micrograph of the ODS 316L in different conditions. Slice 1 was deformed to 5 pct; slice 2 deformed to 13 pct and slice 3, to 25 pct. All images are taken under the same magnification.
V. CONCLUSIONS

1. It has been demonstrated that ODS austenitic stainless steel 316L can be successfully joined to Inconel 718 nickel superalloy by the application of diffusion bonding. The optimal conditions for bonding were found to be 1200 °C for 1 hour, with a decreasing force of initial value 50 kgf applied at the start of the bonding process.

2. Samples that were diffusion bonded in the optimized conditions failed away from the bond line in all mechanical tests, which suggest that diffusion bonding is a promising joining method for ODS alloys.

3. A thermo-mechanical treatment (TMT) based on 25 pct cold rolling, followed by annealing at 1150 °C for 0.5 hour and air cooling, was successful in refining the microstructure of the ODS 316L after diffusion bonding.

4. The nanometric oxide particles were not observed to experience significant change after exposure to the thermal cycle related to the bonding and TMT processes, preserving the size...
distribution found for the as-received ODS 316L alloy.

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**CONFlict OF INTEREST**

The authors declare that they have no conflict of interest.

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