REDUCTION IN DEFECT CONTENT IN ODS ALLOYS

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ABSTRACT

The high temperature creep strength of the oxide dispersion strengthened (ODS) FeCrAl alloys owes much to the development of a suitable grain structure. Existing processing routes may lead to extremely coarse, elongated microstructures which are ideal for creep resistance in specific loading regimes and are made possible by the presence of the oxide dispersion and steered by the route taken during the latter stages of production. For example, in extruded tubes a microstructure elongated parallel to the extrusion direction is usual and has been shown to give enhanced creep performance in applications where the principal stress acts parallel to the elongated grains. In the case of extruded tube intended for use in an internally pressurised system however, the principal stress is a hoop stress and acts perpendicular to the direction of grain elongation, thus substantially reducing the effectiveness of the microstructure. It would seem that the ideal microstructure to resist creep in a pressurised tube where there exist hoop and axial stress components would be a helical one.

The feasibility of creating a helical microstructure in ODS FeCrAl tube by plastically twisting the tube prior to final recrystallisation is presently underway on a laboratory scale and also on a quasi-commercial scale. The greatest obstacle to achieving high levels of torsional strain in a tube is that the tube has a tendency to buckle and collapse. During initial torsion trials a number of experimental parameters, such as temperature, strain rate, degree of twist and the use of mandrels, was investigated. Mathematical modelling of tube collapse is also being performed in tandem. Data collected so far are encouraging and show that a through-wall helically orientated grain structure can indeed be created by the techniques investigated. Twisting trials have now moved on to a larger, more commercially realistic scale using specialist twisting and heating equipment. Aspects of the laboratory, commercial and modelling output will be discussed in this paper.

INTRODUCTION

Alumina-forming ferritic steels with oxide dispersion strengthening (ODS) represent an important alloy group for high temperature applications. They offer the potential for high temperature strength and creep resistance and form a stable, compact α-alumina scale providing excellent high-temperature oxidation resistance. This is combined with the use of cost-effective iron as the main alloy constituent. Not surprisingly, the bulk of the research that has been devoted to the development of these alloys has concentrated on the improvement of mechanical and corrosion properties, and with the potential for increasingly demanding end applications. Research is ongoing to further enhance performance and reliability. Commercial Fe-based mechanically alloyed (MA) ODS alloys such as PM2000, have a composition and microstructure designed to impart creep and oxidation resistance in components operating at temperatures from ~1050ºC to 1200ºC and above. These alloys achieve their creep resistance from a combination of factors including: the dispersion of fine scale (20-50nm diameter) Y_2O_3 particles introduced during MA which, despite formation of complex oxides involving Al from solid solution, is highly stable to Ostwald ripening; and the presence of a very coarse, highly textured, high grain aspect ratio (GAR) structure which results from and is sensitive to the alloy thermomechanical processing history.[1-3] Alloys are, typically, hot consolidated to full density using techniques such as Hot Isostatic Pressing (HIP), extrusion, upsetting or forging following which further hot and cold working (e.g. rolling, drawing etc) is used to produce the final alloy form.[4] Subsequently Fe-based ODS alloys are given a secondary recrystallisation anneal to produce very coarse grain structures for creep resistance.

Despite the benefits offered by the currently processed range of Fe-based ODS alloys they suffer from a number of performance shortfalls. In particular, the high GAR structures induced for creep resistance lead to anisotropic creep properties, which exhibit maximum creep resistance when the principal creep stress is aligned with the major axis of the grain structures.[5,6] But the grain structures evolved during secondary recrystallisation of currently available Fe-based ODS alloys strongly align with the principal product forming direction, which means that in a product such as conventionally HIP'd and hot extruded tube the high GAR direction is along the tube axis.[1,2,5,7] Moreover, in the Fe-based ODS alloys this alignment cannot be altered by directional thermal treatments such as zone annealing.[7] So, for Fe-based ODS alloy tubing currently available for high temperature internally pressurised applications, the direction of maximum creep strength is orthogonal to the direction of maximum principal creep stress (the hoop stress). As a result, creep life in the hoop orientation in Fe-based ODS alloy tube may be no better than 20% of that in uniaxially loaded and crept tube.[8] Moreover, pressurised tube burst data for material with current microstructures indicates a creep life (~ 14,500h / 1100ºC / 5.9MPa pressure) that is ~ 10% of that likely to be required for tube for application in high temperature heat exchangers (100,000h life) for power generation applications.[9]

In a recently completed BRITE Euram project and work which followed on from it, the technique of flow forming was used to produce PM2000 alloy tube products with grain structures with improved grain aspect ratio in the hoop orientation.[10,11] Subsequent secondary recrystallisation of these flow formed tubes resulted in evolution of grain structures that were complex and varied in size, aspect ratio and orientation both as a function of position through the tube wall thickness and with the total level of flow forming deformation applied. These attributes are not necessarily suitable for good high temperature creep performance. In particular, small and variable grain size and aspect ratio and a lack of through-wall grain growth tend to be detrimental to creep properties, except in tubes subject to very high levels of flow forming deformation. With this in mind, other techniques for the manipulation of grain structures in internally pressurised tubes are under investigation. This report details work in the area of one such technique, that of inducing more controlled helical grain structures to develop by means of plastic twisting of the tubes prior to recrystallisation.

DEVELOPMENT OF HELICAL MICROSTRUCTURES VIA TUBE TORSION

The aim of this part of the work was to steer the recrystallised microstructure of ODS PM2000 tubes intended for use in heat exchanger tubing. As the service environment would involve internal pressurisation of the tubes at high temperatures for extended periods, optimisation of the creep properties is important in the direction of the principal stresses. In an internally pressurised tube the principal stress is in the hoop direction with additional axial stress. The ratio of the hoop:axial stresses depends on the inner (ID) and outer (OD) diameters of the tube and is given by $(ID^2+OD^2)/ID^2$. It tends to 2:1 for a thin-walled tube and the optimum microstructure for creep resistance would therefore follow a helical orientation around the tube.

The work described here has been split into three areas; the experiments involving the controlled twisting of PM2000 tubes which took place at ORNL, the recrystallisation studies of the plastically twisted tubes, and commercial trials where long lengths of PM2000 tube were twisted using commercial machinery.

Controlled torsional deformation of PM2000 tubes

In order to steer the recrystallising grains in a helical path, tubes have been twisted along their axis prior to recrystallisation. The twisting was performed at ORNL using an MTS testing machine controlled by MTS Teststar software. This enabled reasonably complex torsional and axial stresses to be applied to the tube with a high degree of control while monitoring relevant parameters. During torsion the tubes were locally heated using a Lepal RF induction apparatus.

The tubes used were 200mm lengths of 25mmOD/20mmID PM2000 tube obtained from Plansee GmbH in the fine-grained condition. PM2000 solid rod of 20mmOD was used as mandrel material for supporting the tube against collapse and against crushing by the test machine grips. The lengths of rod at the ends of the tubes were extended further into the tube than necessary for gripping and were cut to such a length that the gap between them corresponded with the required hot zone. Thus, acting as heat sinks, they helped to constrain the hot zone to a well-defined region of the tube.

The nine tubes tested are shown in Figure 1 after testing and Figure 2 shows a schematic cross-section of a typical tube sample.

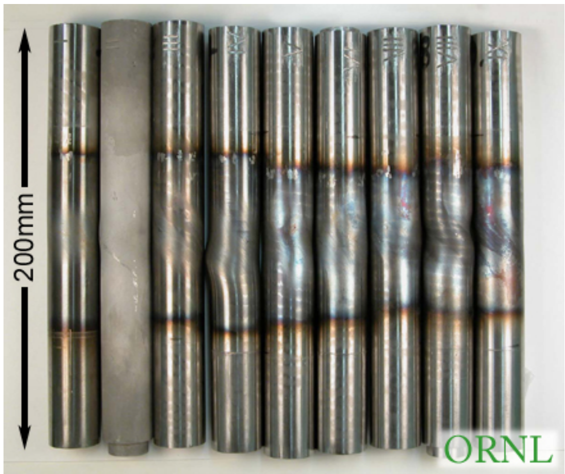


Figure 1
PM2000 tubes after torsion testing

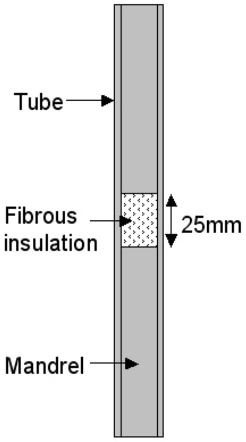


Figure 2
Schematic cross-section of a typical tube

The parameters controlled and recorded during torsion were torque, temperature, total twist, strain rate, axial stress as well as parameters derived from these. Some parameters are shown in Table 1. The significance of the inclination θ and the use of a mandrel in Tube 3 to prevent collapse are illustrated in Figure 3.

Table 1
Torsion test parameters

	Torsion / twisted length [º / mm]	Inclination θ	Temperature [ºC]
Tube 1	83 / 25	36°	750
Tube 2	123 / 25	47°	750
Tube 3[†]	119 / 25	~47°	750
Tube 4	160 / 50	35°	750
Tube 5	80 / 25	35°	750
Tube 6	80 / 25	35°	750
Tube 7	80 / 25	35°	675
Tube 8	160 / 25	55°	750
Tube 9	90 / 25	40°	750

† Tube 3 supported by an internal mandrel

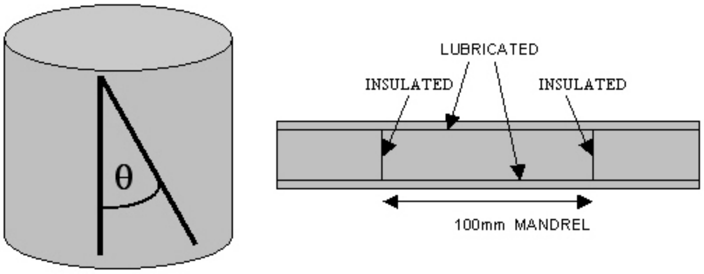


Figure 3
The inclination θ and use of support mandrel.

On the whole, the results of the tube twisting trials were a success. One of the main concerns was whether it would be possible to achieve a significant degree of torsional plastic deformation without the tubes failing plastically. As can be seen from Figure 1, the tubes generally retained their cylindrical form but with degrees of distortion ranging from Tube 8, which showed significant signs of collapse probably due to its high level of deformation (θ=55º), to Tube 3, which was supported by an internal mandrel and showed no signs of distortion at all. It is estimated that Tube 8 started to show clear signs of collapse after twisting through approximately 100º (θ=41º) and this is consistent with the amount of collapse see on Tube 2 (θ=47º) as compared to the much straighter Tube 9 (θ=40º). Tube 5 was subjected to a 16.7kN tensile load during the twisting operation and remained perfectly axisymmetrical but exhibited some axial strain and necking.

Some of the tubes were twisted in 5º increments with a pause of a few seconds between increments while the tubes were checked for collapse. During the pause between twists, the torsional stress developed in the tube during twisting relaxed considerably before resuming its former level when torsion was reapplied. This effect can be seen in Figure 4 from Tube 4, where each small drop in torque corresponds to a pause and the larger drop corresponds to resetting of the grips with the clock stopped. Tubes which were continuously twisted the full amount, with no chance for stress relaxation, seemed to perform better in terms of resistance to collapse. Tube 9 is a good example of this effect and remained very straight despite undergoing a greater degree of torsion than other tubes. It is thought that some work hardening effect may help to stabilise the tubes, stiffening them as required in areas which may be erring towards collapse and thus experiencing more complex and greater amounts of strain. However, if this is the case then it must be a local effect because the overall strengths of Tubes 4 and 6 are very similar and they work harden at similar rates.

The effect of temperature was briefly investigated by twisting one tube (Tube 7) at the lower temperature of 675ºC. It was hoped that on lowering the temperature, work hardening would increase and help to stabilise the tube against collapse. Although Tube 7 showed an increased yield stress compared to Tube 6, as shown in Figure 5, the work hardening rate did not appear to be greatly affected and the tube showed no enhanced resistance to collapse.

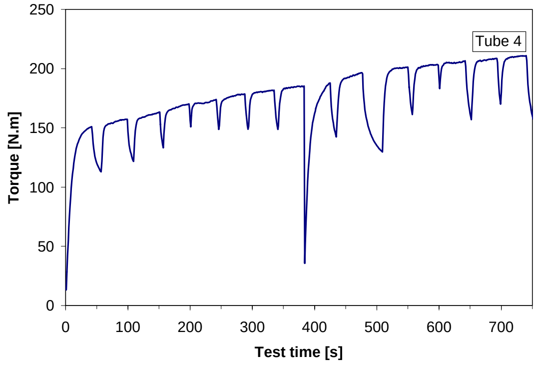


Figure 4
Graph of torque vs. test time for Tube 4.

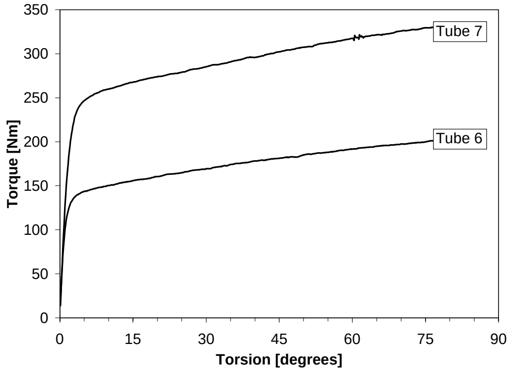


Figure 5
Graphs of torque vs. torsion for Tubes 6 and 7.

Development of helical grain structures in recrystallising PM2000 tube

After twisting, the tubes were prepared for recrystallisation. This involved cutting each tube into six longitudinal segments and then annealing. Tube 4, however, was recrystallised whole. After annealing, the samples were polished to a 1μm finish and etched in 4%HF, 18%HNO_3, 78%H_2O then in Kallings Reagent.

The first tube to be annealed was Tube 4 which had undergone 160° of twist over a length of 50mm. This was annealed at 1380°C/1h in order to fully recrystallise it. The result can be seen in Figure 6. The coarse microstructure in the central, twisted region has clearly been influenced by the twisting process and the grain morphology bears distinct helical features. Although the overall grain shape is still elongated and orientated parallel to the tube axis, the serrated edges of the grains are orientated parallel to the twisted helix. (This is clearer in the higher magnification image in Figure 10.) Figure 7 is a cross section of the tube wall and shows that the coarse grains seen in Figure 7 extend through the entire tube wall.

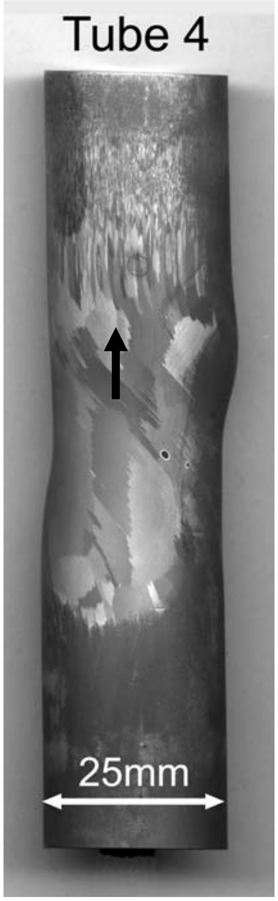
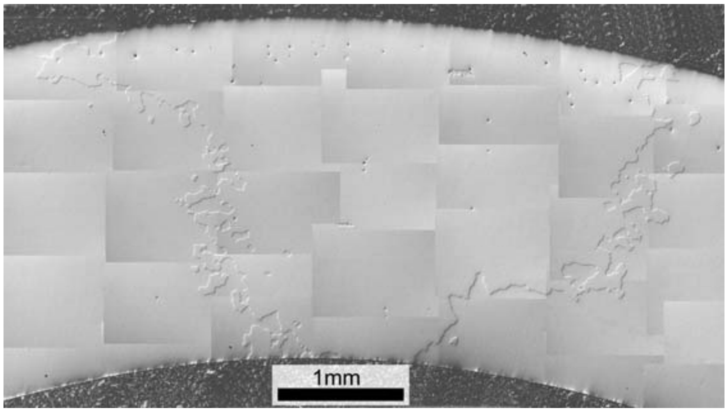


Figure 6
Tube 4 after annealing at 1380°C/1h for complete recrystallisation.
The microstructure in the twisted region exhibits a helical influence but is still axially aligned

Figure 7
A cross section through the wall of Tube 4 shows how grains span the entire wall thickness after recrystallisation.



This may be contrasted to the through-wall microstructures obtained by flow-forming (Figure 8) where no single grain extends through the entire wall thickness. Through-wall grain structures reduce the number of grain boundaries and are therefore beneficial for creep properties.

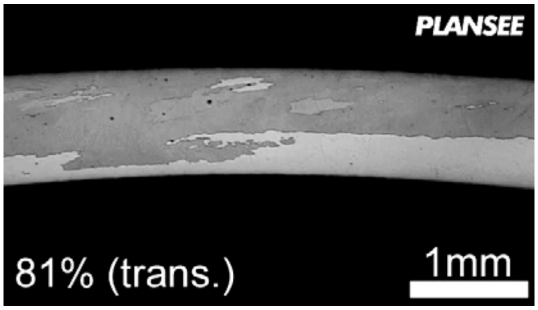


Figure 8
A recrystallised flow-formed tube does not show the same through-wall grain growth as is seen in Figure 7.
This tube had undergone 81% reduction and was annealed at 1380ºC/1h.

Although the ideal microstructure achieved by torsion and recrystallisation would comprise coarse, elongated grains orientated in a helix, the grain structure obtained in Tube 4 still represents a significant improvement on the axially orientated grain structure obtained in recrystallised extruded tube. There are two reasons for this:

- In the untwisted tube oxide particles and other microscopic particulate material are largely distributed in stringers parallel to the tube axis. Such stringers decorate grain boundaries and represent significant lines of weakness normal to the principal hoop stress. In the twisted tube these stringers and the serrated grain boundaries are no longer orientated normal to the hoop stress and therefore would be expected to be less detrimental to creep strength.
- Instead of the planar grain boundaries normal to the hoop stress seen in untwisted tubes, the serrated grain boundaries obtained in Tube 4 are 'knitted' together and orientated largely away from the tube axis, thus providing enhanced strength.

The influence of particulate material within the alloy on the orientation of recrystallised grains can be inferred from Figures 9 and 10. The twisting operation has aligned the particulates in a helical pattern (Figure 9) and it would seem that the migrating grain boundaries of the recrystallising grains are steered by this to follow a similar direction, resulting in the helically orientated grain structures shown in Figure 10. This tube axis is vertical in these figures.

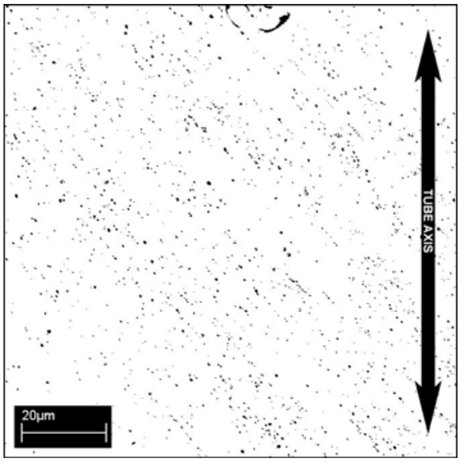


Figure 9
A manipulated image from Tube 4 showing the orientation of particulate material.

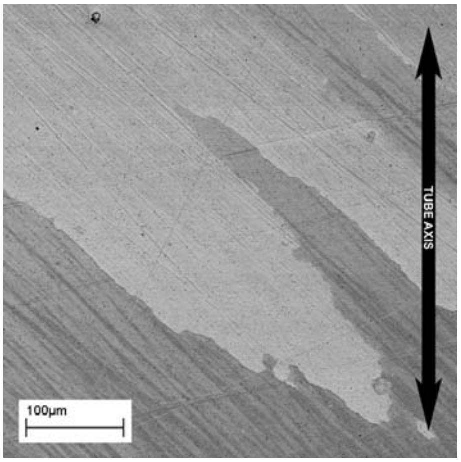
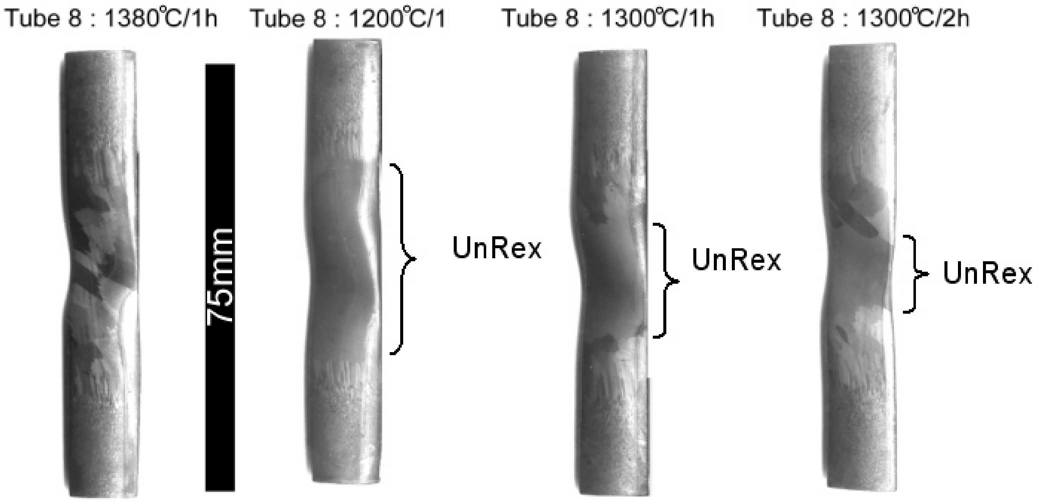


Figure 10
Image of the serrated grain boundaries of Tube 4.

In order to study the effect of greater concentrations of twist, Tube 8 was annealed at 1380°C/1h. Tube 8 had also undergone 160° of twist but over the shorter length of 25mm. The recrystallised microstructure is shown in Figure 11a. The division of the microstructure into fine-grained, untwisted tube and coarse-grained, twisted tube is similar to that seen in Tube 4. The recrystallisation of the torsionally deformed region to produce torsionally aligned large grains may reflect homogenisation of the deformation occurring in the torsion zone at higher deformation, limiting nucleation of recrystallisation and promoting coarser grain structures. This may be similar to the effect found in flow formed tubes, where increasingly large deformations led to increasingly coarse grain structures The coarse-grained region now exhibits an apparently greater helical influence. The grains are generally less elongated parallel to the tube axis and, indeed, several show clear elongation parallel to the helix. There are also several major grain boundaries aligned helically. The greater concentration of twist, as described by the inclination in Table 1, seems to have improved the microstructure and steered it into a more helical pattern.



Figures 11 a-d
Samples from Tube 8 after annealing at different conditions to show the progression of recrystallisation.

The partitioning of the recrystallised microstructures into coarse- and fine-grained regions in the tube prompted investigation of the route by which recrystallisation progressed. To this end, samples of Tube 8 were annealed at temperatures lower than the 1380°C employed for complete recrystallisation. After annealing at 1200°C/1h, the twisted region of the tube had not recrystallised while the fine-grained regions at either side had recrystallised, as shown in Figure 11b. It is not yet fully understood why this should be the case as the twisted region had undergone a greater degree of deformation and might therefore be expected to have a lower recrystallisation temperature. The higher recrystallisation temperature does correspond with the coarse grain size, as would be expected. The possibility that the heating of the twisted region during torsion may have cause recovery, thus reducing stored energy, was tested by annealing a sample at 750°C/15min prior to recrystallisation, but the coarse/fine microstructure was still seen.

Annealing at 1300°C/1h also gave incomplete recrystallisation as shown in Figure 11c, but recrystallisation had progressed further than after 1200°C/1h. On annealing at 1300°C/2h, recrystallisation was still further advanced (Figure 11d) and coarse-grained helical grains had started to grow. It seemed that the recrystallisation might have spread from the extremes of the sample into the twisted centre by oriented, selected growth and this would have had important ramifications for commercial exploitation of this process with long tubes. However, a sample split into three regions prior to recrystallisation at 1380°C/1h exhibited the same coarse, helical microstructures, as can be seem in Figure 12. This suggests recrystallisation by repeated nucleation rather than selective growth.

Figure 12
Split sample used to investigate origins of nucleation.

As previously mentioned, an axial tensile load of 16.7kN was applied to Tube 5 during the twisting operation. As the axial load appeared to have a stabilising effect and helped to retain the tube's axial symmetry, axial load application might be considered as part of a commercial process and it was therefore considered worthwhile to examine final grain structures. As can be seen in Figure 13, the axial load does not seem to have affected the morphology of grain structures formed on recrystallisation and the tube sample still contains the helically-orientated middle section seen in other tubes.

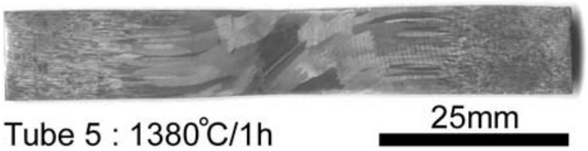


Figure 13
Recrystallised Tube 5 which underwent axial tension as well as torsion prior to recrystallisation.

Initial commercial tube-twisting trials

Having demonstrated that the development of helical grain structures in tubes is feasible on a laboratory scale, the second stage has been to demonstrate whether the technique could be scaled up using a commercially available process.

Forécreu SA, France possess unique facilities for the twisting of long, thin sections under well controlled conditions and agreed to undertake trials involving the twisting of lengths of 25mmOD, 20mmID PM2000 tube. Two 1m lengths were twisted using the apparatus shown in Figure 14. The tubes were inductively heated over a length of approximately 50mm. The size of the hot zone was constrained using cooling air jets. The tube was gripped between two chucks, one fixed and the other rotating at ~2rpm. As the tube was twisted, the hot zone was traversed along the tube at a rate of ~7 mm.s^{-1}.

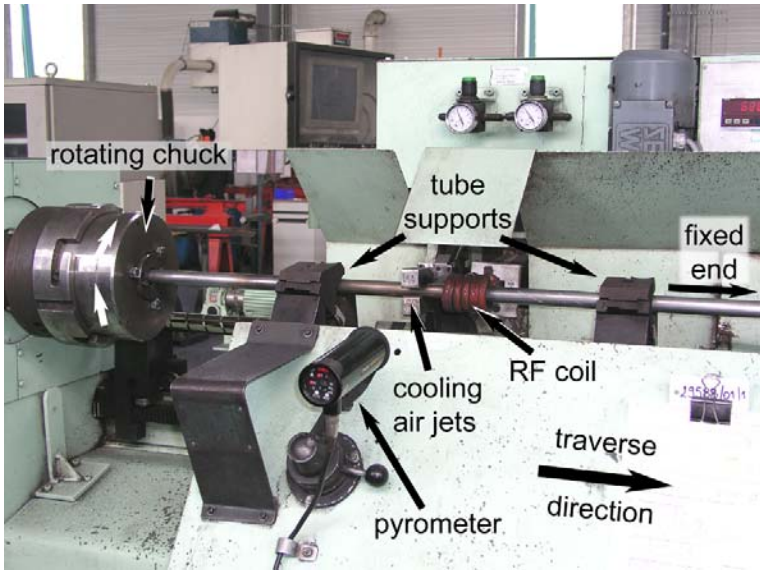


Figure 14
Apparatus used to conduct commercial tube twisting trials.

It was planned to twist the tube to a cumulative θ of 40º in two passes of 20º each along the length of the tube. However, during the first pass of 20º the tube underwent a type of buckling not anticipated. While the section of the tube remained perfectly circular with no signs of the collapse seen in previous laboratory work, a larger scale buckling occurred and the tube became twisted into a spiral shape along its whole length. This can be clearly seen in Figure 15. This type of buckling may be compared to Euler buckling under compression. As the tube is twisted it experiences shortening stresses which lead to instability and buckling. The second length of tube was twisted under a weak axial tensile stress in an attempt to prevent buckling but sufficient stress could not be applied with the apparatus used and buckling occurred again. Two methods are under consideration to address this buckling effect. The first is to apply torsion plus tension to the tube to prevent the buckling taking place. It is not yet known whether a sufficient tensile load to avoid buckling, would cause yielding and axial strain of the tube. An alternative method would be to torsionally deform the tube with the inclusion of a loose-fitting, internal mandrel to limit buckling and to follow the twisting process with a tube straightened procedure. Both methods seem entirely feasible at this stage.

Figure 15
PM2000 tube twisted in commercial trials. Spiral buckling has occurred.

Figure 16
A close-up of the tube shown in Figure 15.

As can be seen from Figure 16, even though buckling occurred, the intended $\theta=20^{\circ}$ of plastic twist was nevertheless introduced to the tube. It is anticipated that helical microstructures will develop on recrystallisation of the tubes and may provide material for larger scale mechanical testing.

CONCLUSIONS

- Tubes of ODS-PM2000 can be successfully twisted on a laboratory scale, introducing high levels of plastic strain into the tube without significant buckling and collapse. An empirical understanding of the process has been developed by the testing of a range of experimental parameters and conditions.

- Torsional deformation has a distinct influence on the microstructure of subsequently recrystallised tube samples. Particulate material within the alloy orientated by the twisting steers grain growth into a helical pattern.

- The helical nature of the grain structure seems to be influenced by the amount of twist prior to recrystallisation. Increased amounts of twist appear to give a greater degree of helicity.

- A method of twisting lengths of ODS tube on a commercial scale had been identified. Initial trials are encouraging and lengths of tubes have been twisted. Methods for countering the unanticipated mode of buckling encountered have already been proposed.

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