Journal of Nuclear Materials 386–388 (2009) 537–539

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Some microstructural characterisations in a friction stir welded oxide dispersion strengthened ferritic steel alloy

F. Legendre [a,*], S. Poissonnet [a], P. Bonnaillie [a], L. Boulanger [a], L. Forest [b]

[a] CEA Saclay, DEN/DANS/DMN/SRMP, 91191 Gif-sur-Yvette cedex, France
[b] CEA Saclay, DEN/DANS/DM2S/SEMT/LTA, 91191 Gif-sur-Yvette cedex, France

A B S T R A C T

The goal of this study is to characterize microstructure of a friction stir welded oxide dispersion strengthened alloy. The welded material is constituted by two sheets of an yttria-dispersion-strengthened PM 2000 ferritic steel. Different areas of the friction stir welded product were analyzed using field emission gun secondary electron microscopy (FEG-SEM) and electron microprobe whereas nanoindentation was used to evaluate mechanical properties. The observed microstructural evolution, including distribution of the yttria dispersoids, after friction stir welding process is discussed and a correlation between the microstructure and the results of nanoindentation tests is established.

1. Introduction

The fusion and fission reactors are faced with important challenges, notably in the field of materials. A class of materials, susceptible to resist the anticipated severe environments, is the ODS family (oxide dispersion strengthened), which are metallic alloys strengthened by a very fine oxide dispersion. The high temperature stability of the fine dispersoids inhibits dislocation motion and leads to an improvement of the creep deformation resistance. An obstacle to the utilisation of these materials is their weldability because conventional fusion welding methods can disturb the fine oxide dispersion in the alloy that leads to a degradation of the high temperature behaviour. Consequently, the friction stir welding (FSW), which is a solid-state joining process, could be an alternative way to weld ODS alloys while keeping the fine microstructure.

The principle of the friction stir welding process consists to insert a rotating pin into the material [1]. The frictional heating and pressure act to plastically stir the material. As the tool is moved forward, the material in the weld zone is mixed and transferred from the leading edge of the tool to the trailing edge, where it cools to form the weld. Since melting does not occur, the material maintains its crystalline properties. The process has been successfully applied to non-ferrous materials such as aluminium [2] but also, by using suitable tool materials, to harder and higher melting point materials such high strength low-alloy steel [3]. However, very few papers report the use of the friction stir welding for ODS steels [4,5].

In the present work, we have investigated the microstructure of a friction stir welded ODS ferritic steel alloy.

* Corresponding author.
E-mail address: flegendre@cea.fr (F. Legendre).

0022-3115/$ - see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnucmat.2008.12.170

2. Experimental

The materials used in this study are an yttria-dispersion-strengthened PM 2000 ferritic steel sheets, manufactured by the Plansee Company. The chemical composition (wt%) is Fe–19Cr–5.5Al–0.5Ti–0.5Y_2O_3.

The friction stir welding step was realised by the DanStir Company. As the goal of this work is to demonstrate feasibility of FSW, the configuration of the friction stir welded product is relatively simple with two PM 2000 ODS sheets laid on top of one another. The welding speed was set at 50 mm/min, and imposed with a gentle ramp from zero up to full-speed. The tool rotational speed during welding was set at 600 rpm in order to secure sufficient heat input and avoid over loading of the tool.

The morphology and the microstructure of the different areas of the friction stir welded material were examined by optical microscopy, scanning electron microscopy (SEM) with a FEG Gemini 1525 from Carl Zeiss, while quantitative atomic analysis and X-ray cartographies were performed using a Cameca SX 50 electron microprobe. Berkovitch nanoindentation (Nanotest 550, Micro-Materials Ltd.) (triangular based pyramidal indenter) tests were performed to investigate the mechanical properties of different areas of the friction stir welded product.

3. Results

The back scattering electron (BSE) image and the X-ray images obtained from the electron probe analysis of the as-received PM 2000 material are collected in the Fig. 1. These analyses show the presence of some micronic precipitates homogenously distributed in the as-received material. Some of them contain titanium, whereas the majority is alumina (Al_2O_3) particles. Concerning the

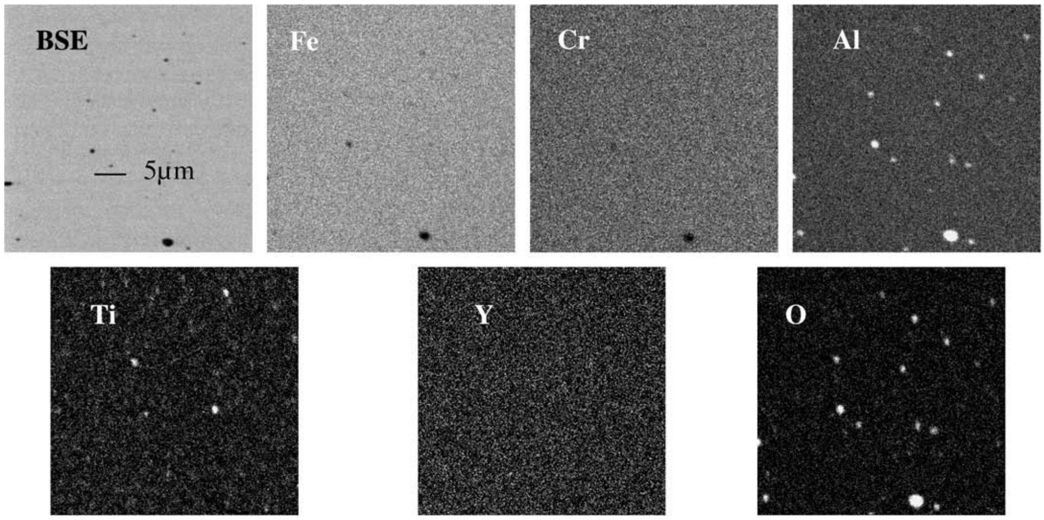

Fig. 1. BSE image and microprobe images of PM 2000 ODS alloy.

yttria dispersoids, the yttrium map is close to the background noise that indicates a homogeneous distribution in the metallic matrix and confirms a submicronic size of the Y_2O_3 reinforcements. Forty nanoindentation tests at 500 nm depth with an applied charge of 30 mN were realised on the base metal. According to the method of Oliver and Pharr [6], indentation load-displacement curves were analysed to calculate the hardness and the reduced modulus. For

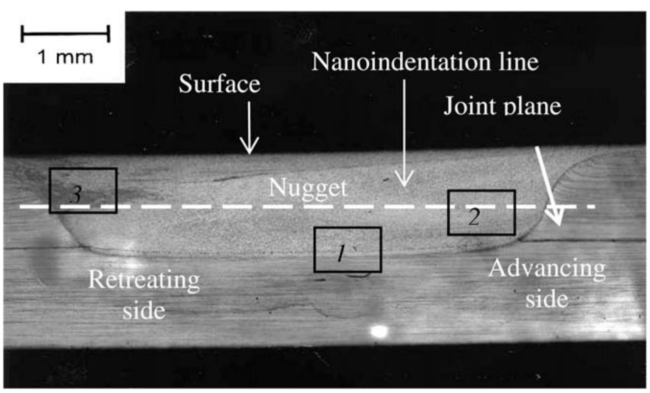

Fig. 2. Optical micrograph of the friction stir welded product.

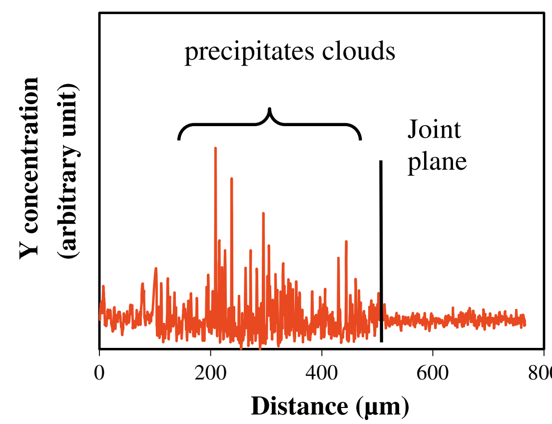

Fig. 4. Yttrium concentration profile along a crossing realised in area corresponding to the zone 2 indicated of the optical micrograph.

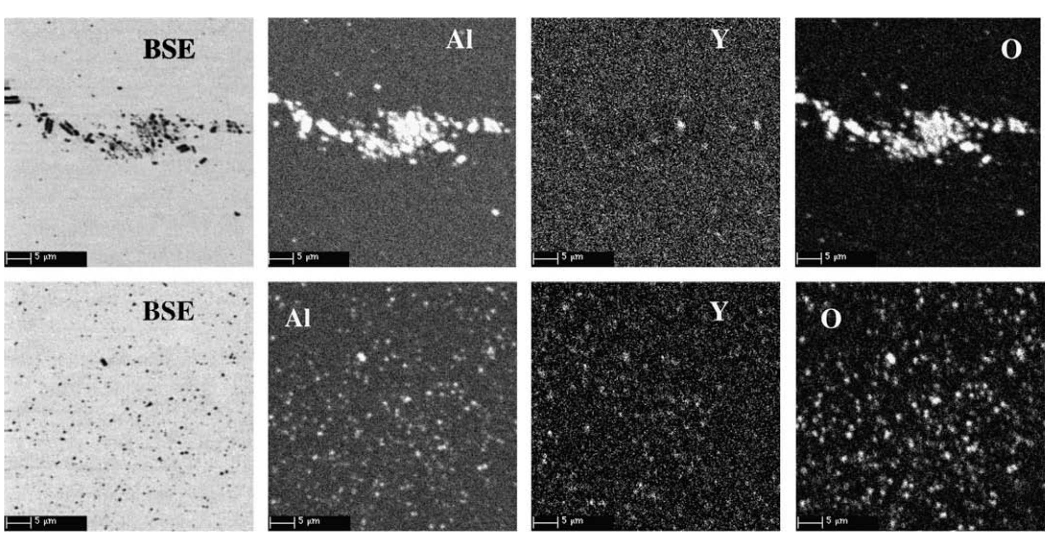

Fig. 3. BSE images and microprobe images of zone 1 (up) and zone 2 (bottom) in the nugget.

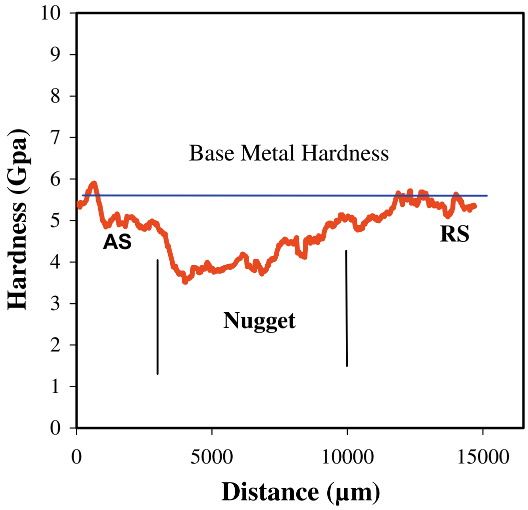

Fig. 5. Hardness profile resulting from nanoindentation experiment conducted parallel to the surface.

the as-received PM 2000 ODS alloy, the average values were 5.4 GPa for the hardness and 210 GPa for the modulus.

Optical micrograph of the friction stir weld product is shown in the Fig. 2. The joint plane is well marked and some clouds of precipitates are visible. One can observe two diametrically opposed areas with large densities of precipitates on the optical micrographs: one near-surface (retreating side) and the other above the joint plane (advancing side). Fig. 3 shows the back scattering electron (BSE) image and the X-ray images obtained from the electron probe analysis of two areas of the nugget zone (indicated by the rectangles 1 and 2 on the optical micrograph). The elemental maps show on the joint plane a high concentration of Al_2O_3 particles with an average size larger than 1 µm. Also, the images show the presence of a small number of Y_2O_3 particles. Above the joint plane, the electron probe images highlight the presence of Y_2O_3 precipitates larger and more numerous than that the ones observed in the base metal. The alumina particles are still present but homogeneously distributed. The different concentration profiles made perpendicular to the joint plane confirm the change in the distribution of precipitates Y_2O_3 in some areas of the nugget. For instance, the graph of the Fig. 4 shows the variation of yttrium concentration in the zone 2 indicated on the optical micrograph. In the same conditions as those used for the base metal, nanoindentation tests were performed on the friction stir welded product. A nanoindentation line, represented in the Fig. 2, was conducted parallel to the surface to 500 µm above the joint plane. The resulting graph, Fig. 5, represents the evolution of hardness. The shape of the curve reflects the change in the microstructure of the friction stir welded material. The hardness evolution is asymmetrical: it decreases rapidly when moving towards the centre of the nugget before increasing continuously thereafter. At either end of nanoindentation line, we find again the average hardness of the base metal. Another nanoindentation line perpendicular to the joint plane was carried out in the zone 2 indicated on the optical micrograph. This experiment highlighted that the average hardness in this nugget zone above the joint plane is close to 4 GPa, a value lower than the one obtained in the ODS base material (5.4 GPa).

Regarding the results of the microstructural characterizations given above, the decrease of the hardness in some areas of the nugget zone can be attributed to microstructure changes, such as the modification of the distribution of the yttria dispersoids but also the evolution of the alumina precipitates. Nevertheless, microscale characterization tools used in this study allow easy evaluation of the changes in the homogeneity of the Y_2O_3 dispersoids distribution but the micronic resolution is not sufficient to quantify an eventual segregation phenomenon in some areas of the nugget. Therefore, further studies are currently underway with the use of techniques well suited at the nanoscale. Results obtained from small angle neutron scattering (SANS) are given by MH. Mathon et al. [7].

4. Conclusions

This study has allowed assessing the evolution of the microstructure, particularly distribution of the precipitates, in a friction stir welded oxide dispersion strengthened alloy. The results have confirmed the feasibility of using friction stir welding process for ODS materials. However, the process leads to some modifications in the dispersoids distribution that should be considered in the future optimisation works.

This study also highlights the potential of using conventional techniques (SEM, electron microprobe and nanoindentation) for microstructural characterization of materials reinforced by nanodispersion, in which the uniformity of the dispersoids distribution determines the mechanical properties. For instance, the electron microprobe allows the monitoring of the homogeneity of dispersion. These tools allow an initial easy characterization before moving on to more sophisticated techniques such as MET, SANS, or Tomographic Atom Probe.

References

[1] R.S. Mishra, Z.Y. Ma, Mater. Sci. Eng. R 50 (2005) 1.
[2] W.J. Arbegast, Weld. J. (2006) 29.
[3] P.J. Konkol, M.F. Mruczek, Weld. J. (2007) 187.
[4] P. Miao, G.R. Odette, J. Gould, J. Bernath, R. Miller, M. Alinger, C. Zanis, J. Nucl. Mater. 367–370 (2007) 1197.
[5] B.K. Jasthi, S.M. Howard, W.J. Arbegast, G.J. Grant, S. Koduri, D.R. Herling, Friction Stir Welding of MA957 Oxide Dispersion Strengthened Ferritic Steel, Friction Stir Welding and Processing III, TMS, 2005.
[6] W.C. Oliver, G.M. Pharr, J. Mater. Res. 17 (1992) 1564.
[7] M.H. Mathon, V. Klosek, Y. de Carlan, L. Forest, J. Nucl. Mater. 386–388 (2009) 475.