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Microstructural and cold workability assessment of a new ODS ferritic steel

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Abstract. A new ODS composition (Fe-14Cr-2W-0.3Ti-0.3Y_2O_3) developed in the ExtreMat integrated Project has been produced by mechanical alloying techniques and consolidated by hot extrusion. This study summarizes some results of characterization and cold workability tests carried out at CEA and EPFL.
It appears that the microstructure is fine and uniform after hot extrusion. According to microprobe analysis, solute elements are homogenously distributed in the matrix. However, the relatively high hardness level measured after hot extrusion and heat treatment may be detrimental in case of additional cold processing which is required to produce final shape like thin plates or cladding tubes. An assessment of the cold workability and the effect of the degree of cold work by rolling on recrystallisation temperature are addressed here. It is found that this material can be successfully cold rolled with a high degree of cold work (up to 60% of thickness reduction) without any damage. According to optical micrographs and Differential Scanning Calorimetry (DSC) measurements, it seems that the recrystallisation temperature remains always very high (above 1400°C) even though cold work level increases (up to 66% of thickness reduction). However, the hardness values begin to decrease for heat treatment temperatures above 1200°C for hot worked conditions and below 1000°C for cold worked conditions, respectively.

Introduction

Oxide dispersion strengthened (ODS) ferritic steels are investigated for structural applications in future nuclear systems. Their use appears attractive because of their high level of mechanical strength up to ~800°C and their good resistance to void swelling [1, 2, 3, 4, 5]. In addition, some alloys can be designed to exhibit low activation properties [2]. To produce finished products like cladding tubes it is important to assess their cold workability. Indeed, the anisotropy of the structure resulting from the extrusion process leads to a lower strength in the transverse direction than in the longitudinal direction and a possible embrittlement during tube fabrication. Previous studies [4, 5] indicate that cracking can be avoided only by using moderate cold work reduction (in the range 15 to 30%) with intermediate annealing in a narrow temperature window allowing to relieve the stress induced by working without recrystallising. For tube manufacturing, two cold-forming processes can be envisaged : cold drawing or HPTR cold rolling. It was shown in [5] that cold-drawing process on ODS steel can induce early cracking from the first steps of the manufacturing.
In this work, some experiments were performed on thin samples by cold rolling. Since the deformation mode in this case is different than in the case of tube manufacturing, other experiments on tube specimens will be planned in a second step. Some characterizations were performed before and after cold rolling and our results are compared to those obtained for MA957 (Fe-14Cr-1W-1Ti-0.3Mo-0.25Y_2O_3) ODS steel [1].

Experimental procedure

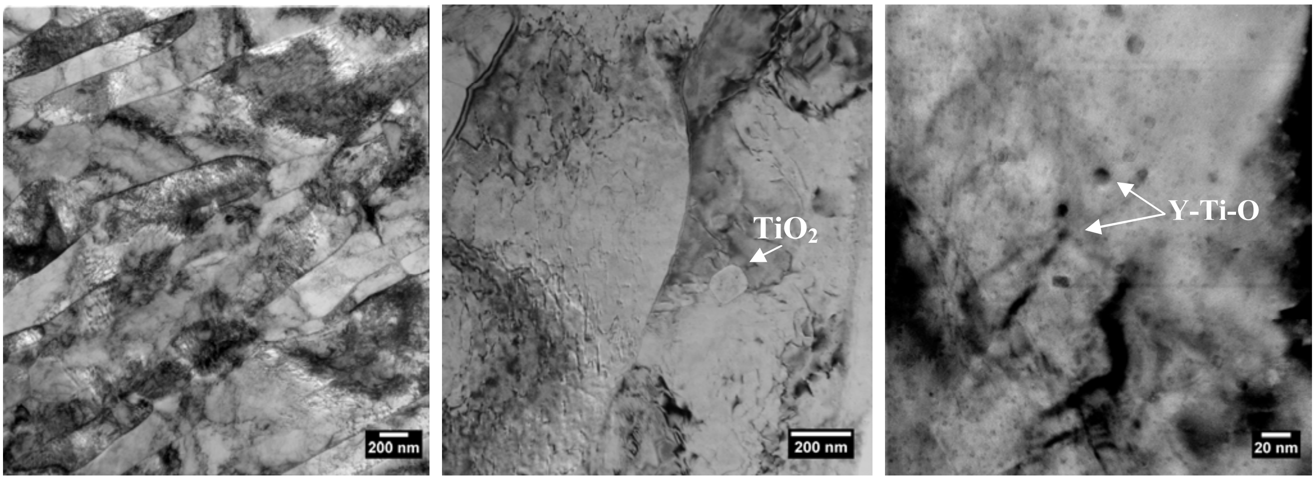
Gas atomized powder of Fe–14Cr–2W–0.3Ti (mass%) produced by Aubert&Duval was mechanically alloyed with Y_2O_3 powder under hydrogen by Plansee by using an attrition type ball mill. The mechanically alloyed powder was then encapsulated in a 75mm diameter soft steel can, and degassed at 300°C. Using a 575 tons press, the cans filled with the mechanically alloyed powder were extruded at 1100 °C into a square section die to get bars with a section of 35x8 mm² (31x5.5mm² on the ODS core). These bars were hot-rolled at 700°C with 20% of thickness reduction and then annealed at 1050°C for 1 hour. The steel can was removed by chemical cleaning.

Some specimens were examined using transmission electron microscopy (TEM) equipped with an energy dispersive spectrometry (EDS) system (point analysis). Coarser particles were observed and identified by means of STEM-EDS detector (mapping of elements). In addition, microprobe analysis was performed to control the distribution of solute elements. Cold rolling experiments were made on specimens of 3mm in thickness by using a reversible rolling mill with a capacity of 3500KN. Heat treatments in static condition at temperatures varying from 1000 to 1450°C were performed under argon atmosphere. Optical microscopy, hardness tests and DSC were used to determine the recrystallisation temperature before and after cold rolling. DSC measurements were carried out by using a SETARAM Calorimeter and a heating rate of 10°C/min.

Results and discussion

TEM examinations and microprobe analysis

TEM examinations reveal a very fine and uniform microstructure with elongated grains in the extrusion direction and equiaxed grains with a size up to a few hundreds of nanometers in the transverse direction (Fig. 1a,b). Some large particles up to 200nm in diameter are identified as TiO_2 in accordance with EDX analysis. Y-Ti-O nanoclusters are also observed with a size less than 10 nanometers in diameter (Fig.1c). The large density of dislocations made the observation of these yttria nanoparticles difficult.



a) b) c)

Figure 1. Transmission electron micrographs of the ODS steel after consolidation and heat treatment 1050°C-1h showing the microstructure and the precipitates a) longitudinal direction b) c) transversal direction

Composition profiles were measured with an electron microprobe on a transverse cross-section of the square-section bar. The measurements were carried out step by step of 1 µm each operating over a distance of 1mm. As shown in figure 2a, this new alloy exhibits a low oxygen content (< 1000ppm) which is consistent with a chemical analysis (O ~ 914ppm). The titanium profile shows some peaks which may be attributed to the presence of precipitates in agreement with optical and TEM examinations. Measurements of the tungsten profile indicate a homogeneous concentration with an average value of around 1.75 (in weight %) slightly below the expected nominal content (2%). The yttrium profile appears to be homogeneous without depleted zones. This kind of result is not observed for ODS steel obtained by using elemental powders as starting material [7]. The yttrium measured values are varying in the range of 0.18 – 0.30 in weight % with an average value of 0.24%, which is also consistent with the expected composition (Fig. 2b) .

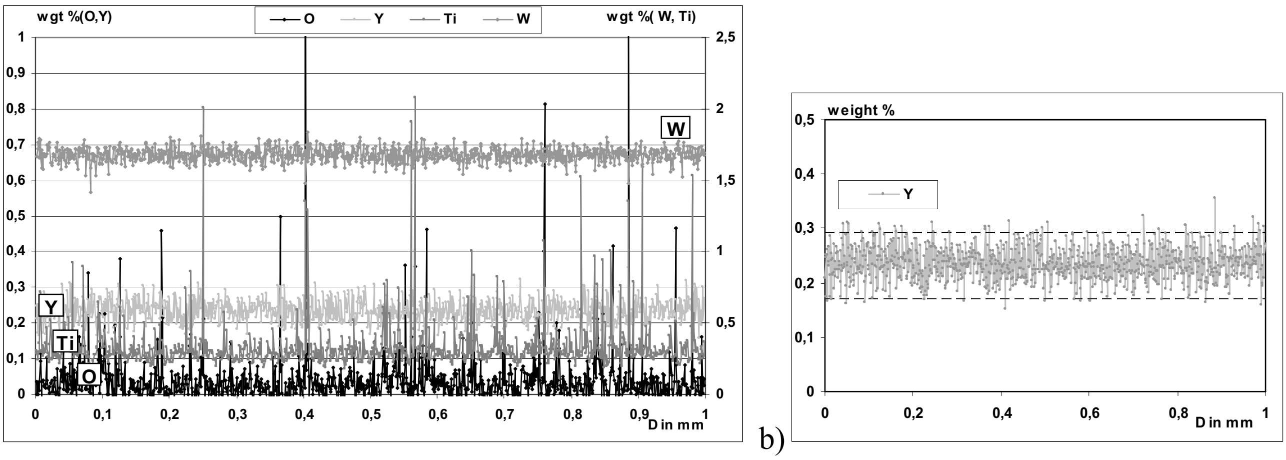
a)  b)

Figure 2. a) Composition profiles for Y, W, Ti, O obtained by electron microprobe b) Y only

Cold-workability and recrystallisation behaviour

The cold-workability of this ODS steel was investigated by cold rolling tests on sheet samples of 3mm thickness. It is found that this material can be successfully cold rolled with a high degree of cold work (up to 60% of thickness reduction) without any damage. For a higher degree of cold work (ε = 66%) cracking occurs. The hardness increases gradually with the level of cold work (Fig. 3).

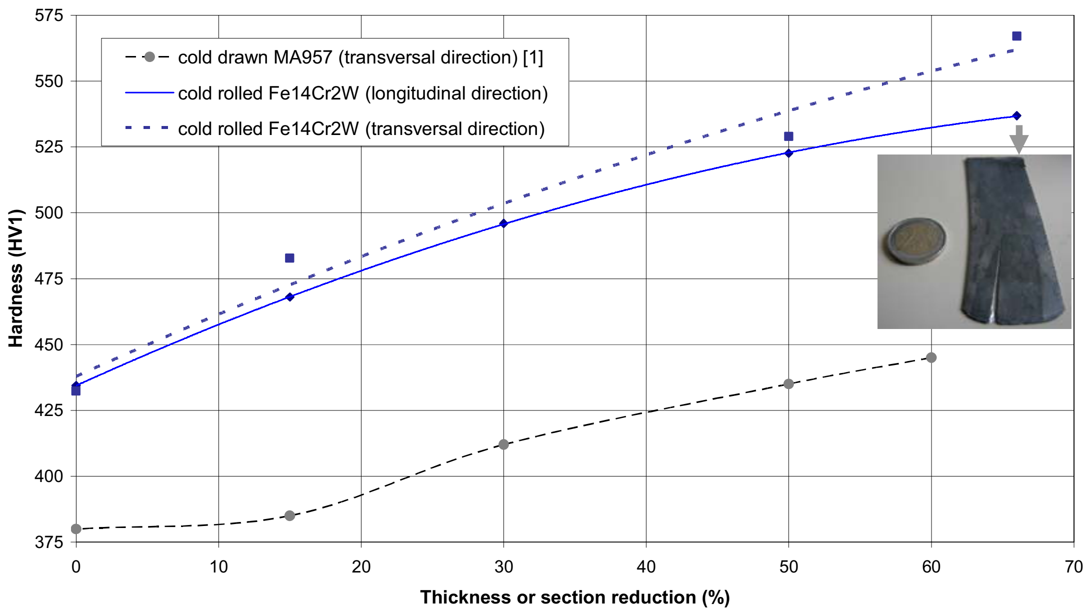
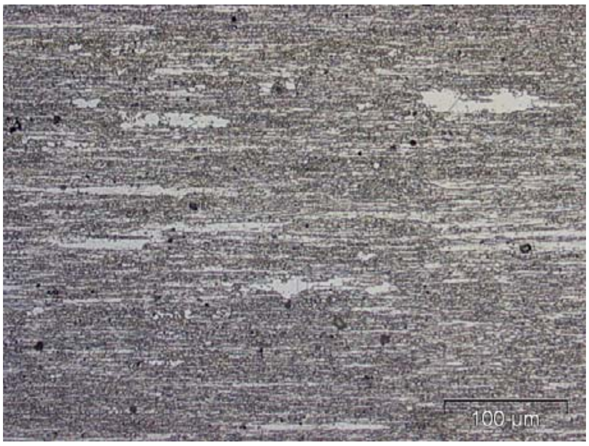


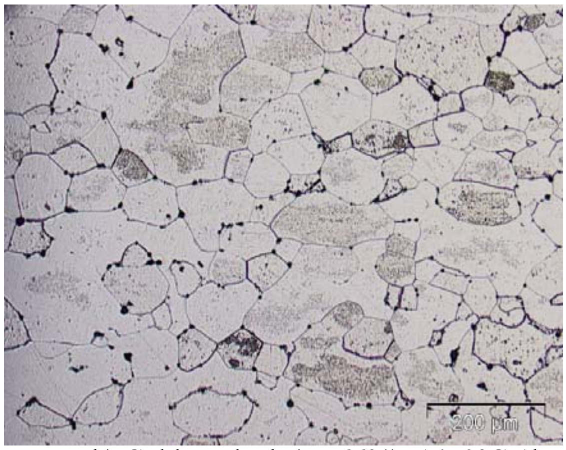
Figure 3. Hardness measurements as a function of cold work reduction for ODS steels obtained by cold rolling (Fe14Cr2W) or by cold drawing (MA957) [1]

It must be noticed that both initial and final values are significantly higher than those obtained for cold drawn MA957 ODS bars for the same thickness or section reduction [1]. The use of a square section die for extrusion instead of a more conventional round section is expected to be mainly responsible for this high level of hardness [4]. In addition, the presence of 2% tungsten in the matrix and some changes in mechanical alloying and hot working parameters may also influence the hardness.

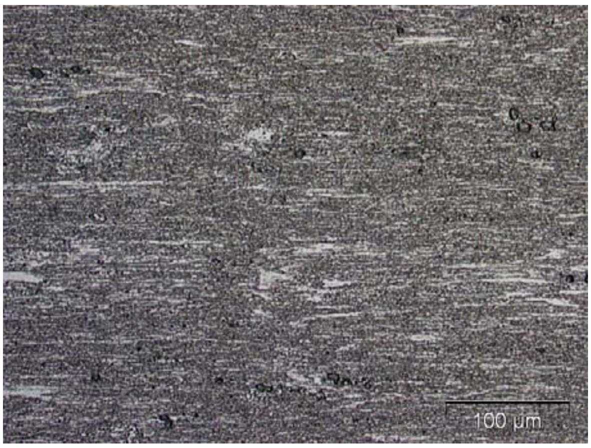
The effect of the cold work reduction by rolling on the recrystallisation temperature was studied by using optical microscopy and hardness measurements in longitudinal direction before and after heat treatments up to 1450°C. In addition DSC experiments were carried out up to 1400°C.
As shown in figure 4d, it appears that the recrystallisation temperature is below 1450°C for as-extruded condition due to the fine and stable dispersion of nanoparticles. This value is similar to what was obtained for the MA957 ODS steel [1].
It seems that the recrystallisation temperature remains always very high, even though the cold work level increases up to 66%. By observing the microstructures obtained after a heat treatment at 1350°C (figures 4a and 4c), it appears that only a few elongated grains begin to grow. This might be due to an abnormal grain growth which leads to a bimodal grain size distribution as observed in the MA957 ODS steel in [1] (the misoriented grains grow first in fine grained matrix because their boundaries move easily). After a heat treatment of 1 hour at 1450°C, the material is completely recrystallized and the grain boundaries are impinged by particles (figures 4b). However, the size and the morphology of the recrystallised grains are noticeably modified in the case of prior cold working (fig. 4b) compared to hot worked condition (Fig. 4d).



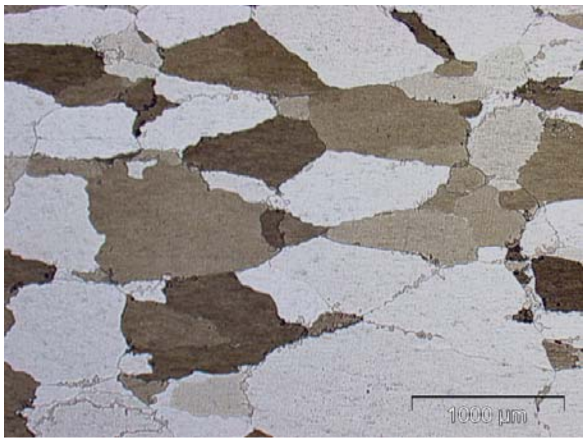
a) Cold worked (ε =66%) -1350°C-1h



b) Cold worked (ε =66%) -1450°C-1h



c) Hot worked -1350°C-1h



d) Hot worked -1450°C-1h

Figure 4 : Optical micrographs showing the different microstructures after heat treatment at 1350°C or 1450°C for hot worked or cold worked samples (ε =66%)

DSC curves do not indicate clearly the presence of any exothermic peak between 1000 and 1400°C for two consecutive cycles from 20°C up to 1400°C (with 10min holding time) for hot worked as well as for cold worked conditions (see in figure 5 the curves obtained for a sample with 66% of CW). This result tends to confirm that the complete recrystallisation occurs only at a temperature above 1400°C in both cases.

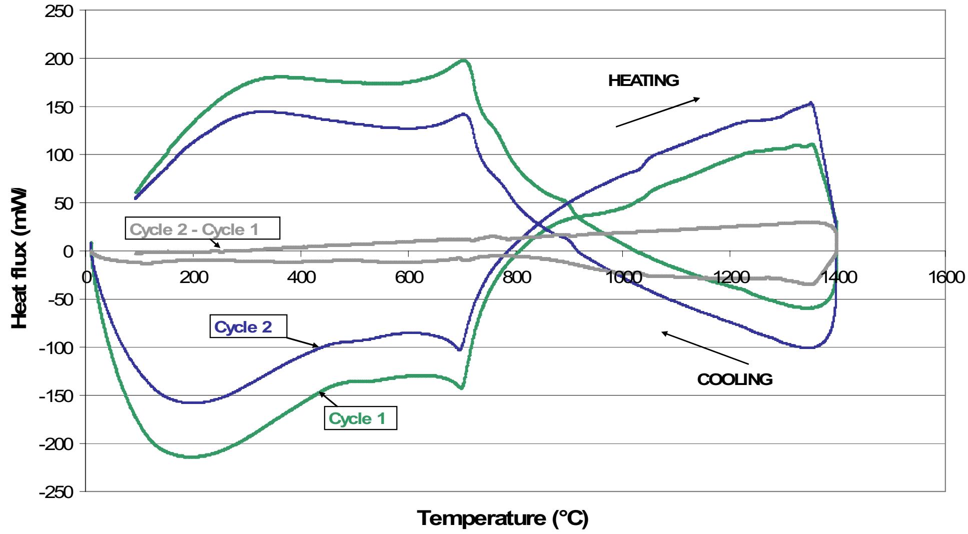


Figure 5. DSC curves obtained for a cold worked sample (ε =66%) during two consecutive cycles from 20°C up to 1400°C

However, an increase in the annealing temperature from 1000 to 1450°C results in a decrease of the hardness values (Fig. 6) for hot worked as well as cold worked samples. The hardness values begin to decrease for heat treatment temperatures above 1200°C for hot worked conditions and from 1000°C for cold worked conditions (66% of thickness reduction), respectively. Above 1200°C, the same hardness values are obtained in both cases which could indicate that all the dislocations created by cold working were eliminated when this temperature is reached.

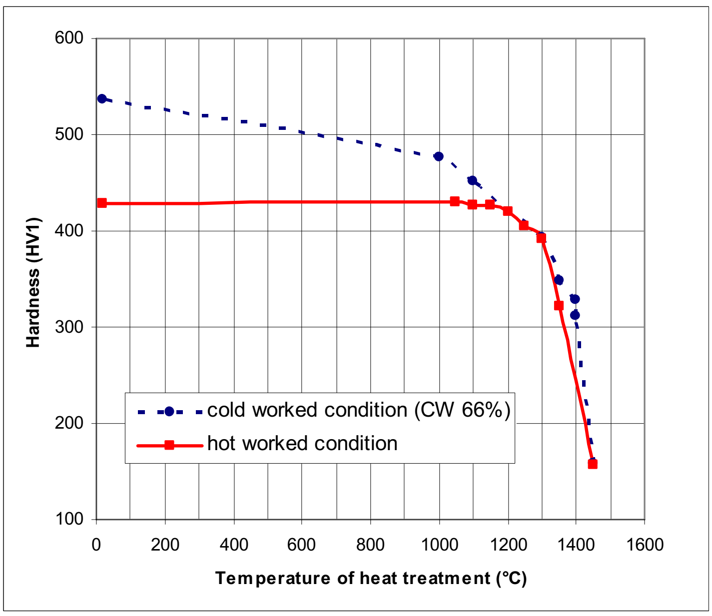


Figure 6. Effect of the heat treatment temperature on the hardness of samples with or without prior cold rolling (for one hour of heat treatment)

These results suggest that a primary recrystallisation can occur for a domain of temperatures in the range of 1100-1400°C as it is mentioned in [1]. During this primary recrystallisation, it is possible that the grain size was only slightly modified at a sub-optic scale. Indeed, as the initial grain size is very fine (around a few hundred of nanometers according to fig. 1), the sub-grains which are formed after elimination and/or rearrangement of dislocations can not move over a long distance due to the large number of original grain boundaries.

Summary

The new ODS-Fe14Cr2WTiY_2O_3 produced in this study exhibits after consolidation a very fine and uniform microstructure and the yttrium profile appears to be homogeneous without depleted zones.
It is found that this ODS alloy can be successfully cold rolled with a high degree of cold work (up to 60% of thickness reduction) without any damage.
According to optical micrographs and DSC measurements, it seems that the recrystallisation temperature remains always very high (above 1400°C) even though the cold work level is increased significantly. However, as the hardness values begin to decrease for lower heat treatment temperatures, a primary recrystallisation should occur in this ODS steel for a domain of temperatures in the range 1100-1400°C.
It appears that cold rolling process does not modify notably the recrystallisation temperature when the cold work level is increased up to 66%. In contrast, by referring to results obtained in [1] for the MA957 ODS steels deformed by cold drawing it is clear that this last process leads to a decrease of the recrystallisation temperature of several hundred degrees by increasing the cold work level from 30% up to 60%. Due to the sensitivity of this kind of material to the deformation process, further experiments on tubes deformed by cold drawing or HPTR rolling would be needed, in order to define a processing window for tube manufacturing (cold work level, interpass anneals) which allow this new ODS steel to recover its ductility (hardness decreasing) by saving the fineness of the microstructure.

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