MECHANICAL PROPERTIES OF Fe-Cr-Al-Si-Ta ODS STAINLESS STEELS

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Abstract. Mechanical alloying was employed to develop new ODS ferritic stainless steels. The mechanical alloying behaviour and consolidated mechanical properties of the Fe-14Cr-2Al-1Si-0.3Ta-1Y $_2$ O $_3$ ferritic steels were strongly influenced by processing parameters. A comparison made with MA 957 and DY-01 alloys showed that the mechanically alloyed Fe-14Cr-2Al-1Si-0.3Ta-1Y $_2$ O $_3$ ferritic stainless steel have interesting properties to be exploited as high temperature materials.

1. INTRODUCTION

Ferritic stainless steels have recently attracted much attention for various high temperature applications such as cladding materials for the Generation IV nuclear reactors [1,2] and interconnects for oxide fuel cells [3,4]. They are expected to have a better swelling resistance and creep strength under high neutron irradiation than austenitic steels. However, conventional ferritic stainless steels have insufficient mechanical properties at high temperatures due to the coarsening of carbides. Advanced ferritic steels without carbon addition have been thus developed, employing strengthening by oxides instead of the precipitation-hardening of carbides. For this purpose, mechanical alloying (MA) is one of the most effective methods to uniformly disperse nano-sized oxide particles such as yttria(Y₂O₂) in the matrix. This technique can be also employed to fabricate nano Fe alloys without the oxide dispersion [5]. Using MA, we have fabricated a new ODS ferritic steel, Fe-14Cr-2Al-1Si- $0.3\text{Ta-1Y}_2\text{O}_3$ (wt.%, named as the alloy ANU-1 in this paper). A comparison was made with commercial MA 957 and DY-01 which was developed in Belgium for the Liquid Sodium Fast Reactor [6].

2. EXPERIMENTAL PROCEDURE

The elemental powder mixture with the nominal composition of Fe-14Cr-2Al-1Si-0.3Ta-1Y₂O₃ (wt.%) was mechanically alloyed using a Szegvari Attritior (Model 1S). The milling balls were made of hardened martensitic stainless steel with a diameter of 6.4 mm. Ball-to-powder weight ratio was 20:1. The speed of rotating arm was 380 r.p.m. The milled powders were packed into mild austenitic stainless steel cans and evacuated to less than 10⁻² Pa for an hour at 450 °C, followed by sealing. The cans were extruded at 1050 °C with an extrusion ratio of 12:1. The core parts of Fe-14Cr-2Al-1Si-0.3Ta-1Y₂O₃ steel were cold-rolled to 1 mm thickness with intermediate annealing at 1050 °C for 1/2 hour. For comparison, some powders were consolidated by HIP (hot isostaic pressing) at 1050 °C at a pressure of 500 bar. Two reference alloys, MA957 (Fe-14Cr-0.3Mo-1.0Ti-0.25Y₂O₃) and DY-

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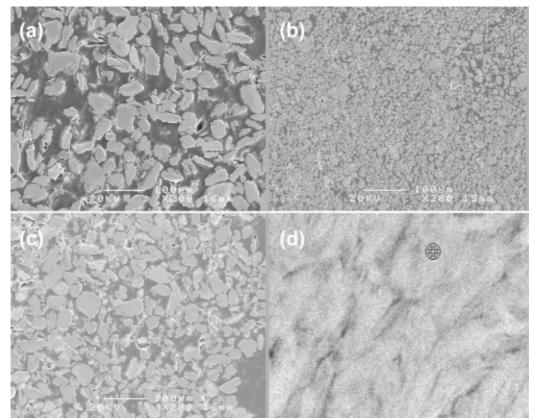


Fig. 1. Powder morphology: (a) MA957, (b) DY-1 and (c) ANU-1 after mechanical alloying for 30 hours. (d): EDXanalysis image of the ANU-1 powder after milling for 30 hours.

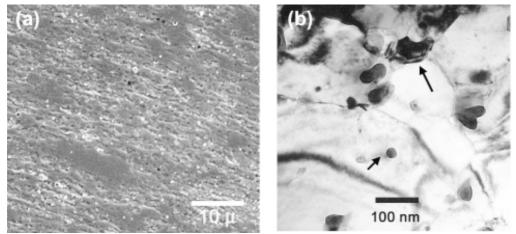


Fig. 2. Micrographs of ANU-1 after hot extrusion: (a) SEM image in the longitudinal direction and (b) TEM image.

01(Fe-13Cr-1.5Mo-3.5Ti-2.0 $\rm TiO_2$ -1.0 $\rm Y_2O_3$) were prepared under the same conditions.

3. RESULTS AND DISCUSSIONS

Fig. 1 shows the powder morphology of MA957, DY-1 and ANU-1 after mechanical alloying for 30

hours. The ANU-1 powder has the mean particle size slightly smaller (~ 12 μm) than the MA957 (17 μm), but larger than DY-01(10 μm). A larger amount of oxides in DY-1 (2.0 wt.% TiO $_2$ and 1.0% Y_2O_3) resulted in finer particle size after ball milling, compared with MA957 (0.25% Y_2O_3) and ANU-1(1.0%

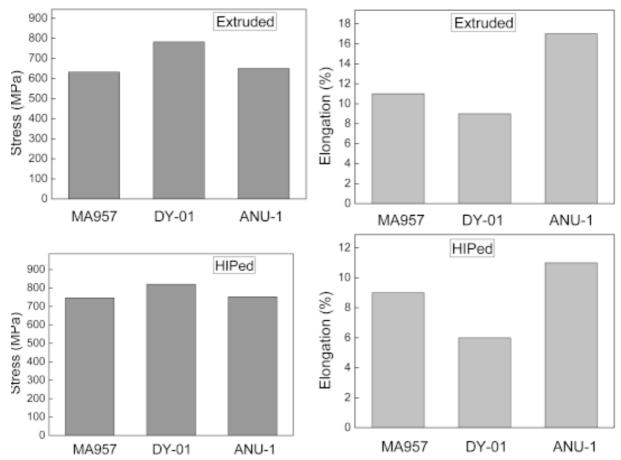


Fig. 3. Comparison of tensile strength (UTS) and elongation values, tested at 500 °C (4·10⁻⁴sec⁻¹) for three alloys prepared under the same processing conditions.

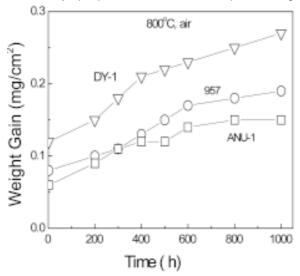


Fig. 4. Oxidation behaviour of three alloys as a function of exposure time in air at 800 °C.

Y₂O₃). The results of EDX showed that the constituent elements of the ANU-1 powder were rela-

tively well distributed after milling for 30 hours. (Fig. 1). The dark parts in the microstructure correspond to slightly Cr-rich regions than the matrix phase.

High temperature consolidation of these alloys was difficult due to their body centered cubic structure inherent to the ferritic steels. Full density was obtained after both HIP and hot-extrusion. HIPed specimens have equiaxed grains (5~10 μ m). Fig. 2 shows the micrographs of the ANU-1 alloy in the longitudinal direction after hot extrusion. The grains in the micrographs are strongly elongated along the direction of extrusion. In spite of preheating and hot extrusion at 1050 °C, the nanoscale size of Y_2O_3 particles produced by mechanical alloying apparently maintained their size. (arrow labes in Fig. 2b) The TEM/EDS analysis showed that the Y_2O_3 particles as well as Al-Si-Y-O based complex oxides with a particle size between 10~60 nm were dis-

tributed not only in the *b.c.c*-structured matrix grains but also along the grain boundaries.

Fig. 3 shows the summarized results of tensile test measured at 500 °C for three alloys prepared under the same processing conditions. Both for extruded and HIPed specimens, the DY-1 exhibited a higher strength and lower ductility than MA957 and ANU-1 due to higher content of oxide dispersion (1% Y₂O₂ + 2% TiO₂). However, the DY-1 was more readily softened by subsequent annealing at 1200 °C for 1/2 hour. It would appear that DY-1 has been overly alloyed with less uniform microstructures with a probable formation of coarsened Y2TiO5 by dissolution of the Y2O3 dispersoids. The ANU-1 exhibited similar or slightly higher strength values than MA957. However, the ANU-1 has much better oxidation resistance at 800 °C than DY-1 and MA957 (Fig. 4). This may be attributed to the presence of Al and Si which play a beneficial role to minimize oxidation by the formation of Alor Si-based protective amorphous layer on the surface of the alloys. The constituent elements of DY-1 and MA957 such as Mo and Ti which are not included in the ANU-1 might be more susceptible to oxidation by gaseous phases at elevated temperatures.

In summary, we have prepared a new ODS ferritic steel, Fe-14Cr-2Al-1Si-0.3Ta-1Y₂O₃ by mechanical alloying and subsequent hot-consolidation. The preliminary result shows that this alloy has high temperature strength comparable to MA957 with much better oxidation resistance at high temperatures. The constituent elements Al and Si in this

alloy facilitate the formation of Al-Si-Y-O based oxide nanoparticles. These oxides as well as nanosized Y₂O₃ produced by mechanical alloying strengthened the alloy with further possible solution hardening by Ta. They are also beneficial for the oxidation resistance at high temperatures.

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