

**DEVELOPMENT OF OXIDE DISPERSION STRENGTHENED FERRITIC STEELS FOR FUSION -  
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### **OBJECTIVE**

The objective of this research is to develop low-activation oxide dispersion strengthened fully ferritic steels for first wall applications in a fusion reactor.

### **SUMMARY**

Seven ODS steels, Fe-(5-13.5)Cr-2W-0.5Ti-0.25 Y<sub>2</sub>O<sub>3</sub> (in weight percent) were manufactured using the mechanical alloying process. Only the composition Fe-13.5Cr-2W-0.5Ti-0.25Y<sub>2</sub>O<sub>3</sub> showed no austenite formation at any temperature using differential thermal analysis and hence was selected as an experimental alloy for the present investigation. Milled powders were consolidated by hot isostatic pressing and hot swaging. Electron microscopy studies indicated high material homogeneity. The hardness of the as-swaged specimen was 65 R<sub>c</sub>. Annealing of the as-swaged material at 800°C, 900°C, 1000°C, 1100°C and 1200°C showed a minor decrease in the hardness.

### **PROGRESS AND STATUS**

#### Introduction

The oxide dispersion strengthened (ODS) ferritic steel called MA 957,<sup>1</sup> produced by mechanical alloying, has received international consideration for fuel cladding applications in liquid metal fast breeder reactors.<sup>2</sup> The alloy shows excellent long term microstructural stability in irradiation environments<sup>3</sup> and is expected to retain superb high temperature strength. This MA 957 alloy has the composition Fe-14Cr-1Ti-0.25Mo-0.25Y<sub>2</sub>O<sub>3</sub>. The microstructure consists of a metal matrix with uniformly distributed Y<sub>2</sub>O<sub>3</sub> dispersoids on the order of 5 nm in diameter. It also contains a highly elongated subgrain structure which is introduced by thermo-mechanical processing. Based on the performance demonstrated to date, this technology should be considered for first wall applications of a fusion reactor.

Therefore, an effort has been initiated<sup>4-5</sup> to consider the use of mechanically alloyed ODS alloys for fusion by altering the alloy composition to be in line with low activation criteria. The objective of the present investigation was to continue that development by optimizing the composition and determining the creep response of the ODS alloy. The alloy composition selected was Fe-13.5Cr-2W-0.5Ti-0.25Y<sub>2</sub>O<sub>3</sub>.

#### Experimental Procedure

The starting powder compositions and the maximum mesh sizes are shown in Table 1. A Spex 8000 shaker mill was used for the mechanical alloying. The alloy was optimized by studying the phase transformations of the powders which are mechanically alloyed and then annealed at 1000°C for 1 h, using a Perkin-Elmer DTA 7 differential thermal analyzer (DTA). The optimization of the

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composition was achieved by studying seven ODS steels, Fe-(5, 9, 10, 11, 12, 13, 13.5) Cr-2W-0.5Ti-0.25Y<sub>2</sub>O<sub>3</sub>, produced by mechanical alloying. The milled powders were also characterized in a Siemens D 5000 X-ray powder diffractometer to understand the structural evolution during milling. Low carbon steel tubing with 1" ID x 0.14" wall thickness x 13" overall length was used for HIP canning. The cans were pumped down to 20 millitorr and then baked at 400°C for 30h and heat crimped before HIPping. The hot isostatic pressing was then carried out on at 950°C and 210 MPa by IMT Inc., Andover, MA. The outside diameter of the HIPped billets (including the can) was 7/8".

After soaking at 950°C for 1 h following HIPping the billets were hot swaged in a hot swaging unit. The outer diameter of the hot swaged materials was 1/2". The cans were opened after hot swaging. The inner diameter of the ODS material was found to be 5/16".

Hardness measurements of the ODS compacts were done in a Tukon hardness tester. Thin foils were made from the compact and characterized in JEOL 1010 and 2010 F transmission electron microscopes (TEM).

The chemical assay of the milled powders was done by Crucible Research, Pittsburgh, PA.

Table 1: Powder specifications

Powders	Size (mesh)	Compositions
Fe	-325	99.4% pure
Fe-Cr master alloy	-200	Fe-73.53 wt.% Cr
Fe-Ti master alloy	-100	Fe-40.53 wt.% Ti
W	-200	99.9% pure
Y <sub>2</sub> O <sub>3</sub>	-325	99.9% pure

## Results

### 1. Alloy Optimization

The x-ray diffraction patterns of all the seven alloys indicated the presence of only the  $\alpha$ -Fe solid solution after 10 h of milling. Figure 1 shows the x-ray diffraction patterns of the Fe-13.5Cr-2W-0.5Ti-0.25Y<sub>2</sub>O<sub>3</sub> (ODS 13.5Cr) powder as a function of milling time. All seven ODS steel powders produced by mechanical alloying were annealed at 1000°C for 1 h prior to the phase transformation studies in a DTA. The DTA plots of the ODS steel powders for 5, 13 and 13.5 Cr milled for 10 h (Figure 2) indicates no austenite transformation only in the ODS 13.5 Cr steel. The amount of interstitial elements in the ODS 13.5 Cr steel powder milled for 10 h was obtained by chemical analysis and was given as follows:

carbon : 0.055

oxygen : 0.855

nitrogen : 0.284

### 2. Characterization

Metallographic sections of the as-swaged material in both the transverse and longitudinal directions were prepared and the results are shown in Figure 3. The micrograph reveals the elongated grain structure in the longitudinal direction. No porosity was observed in the material. A calculation of the bulk density from the machined dimensions showed the material to be 100% dense. The hardness of the as-swaged material was measured to be 65 R<sub>c</sub>. The TEM micrograph in Figure 4 shows the

elongated grain structure and also the presence of very fine dispersoids of sizes less than 10 nm in the ferrite matrix.

The as-swaged material was annealed at 800°C, 900°C, 1000°, 1100°C and 1200°C for 1 h in order to examine the change in the hardness with annealing temperature. A plot of hardness versus annealing temperature in Figure 5 showed a slight decrease in the hardness value with an increase in the annealing temperature. The plot in Figure 5 clearly indicates that the hardness remains very high even following a 1200°C anneal.

## Discussion

### 1. Homogeneity of the $\alpha$ -Fe Solid Solution

X-ray diffraction of the as mixed powders showed only reflections of Fe and W. The Cr and Fe reflections superimpose since their lattice parameters are very close. The amounts of Fe-Ti and  $Y_2O_3$  powders were so small that they could not be detected in the x-ray pattern of the as-mixed powder. X-ray diffraction of the powder milled for 10 h showed the presence of only  $\alpha$ -Fe, with a lattice parameter of 2.89 Å (calculated from the (110) reflection). The disappearance of W reflections and the increase in lattice parameter from 2.86 Å to 2.89 Å demonstrates that W was in solid solution following mechanical alloying. The chemical analysis of the powder obtained from the analytical TEM matches with the targeted composition.

The variations in composition studied covered 5-13.5 Cr. It can be noted that in the pure Fe-Cr system the austenite loop ends at a composition of 12.7 Cr. The presence of 2% W and 0.5% Ti in the  $\alpha$ -Fe solid solution should further suppress the austenite loop to about 10.75 Cr (calculated from chromium equivalent formula<sup>6</sup>) and hence the transformation to austenite should not be observed in the alloys with Cr > 10.75 %. However, the austenite formation was observed by DTA in the ODS (11, 12, 13) Cr alloys. The presence of nitrogen and carbon which act as austenite stabilizers are probably responsible for the austenite transformations in ODS (11, 12, and 13) Cr alloys.

### 2. Microstructural Stability

The as-swaged material shows an elongated microstructure both in optical and transmission electron microscopy typical of hot worked ferritic ODS alloys. Analytical microscopy also indicated high homogeneity in the material. The hardness of the swaged material was very high due to the high dislocation density in the material. High hardness value was retained after 1 h exposure of the as-swaged material at 1200°C indicating a high thermal stability of the microstructure. Therefore, we can anticipate excellent high temperature creep response.

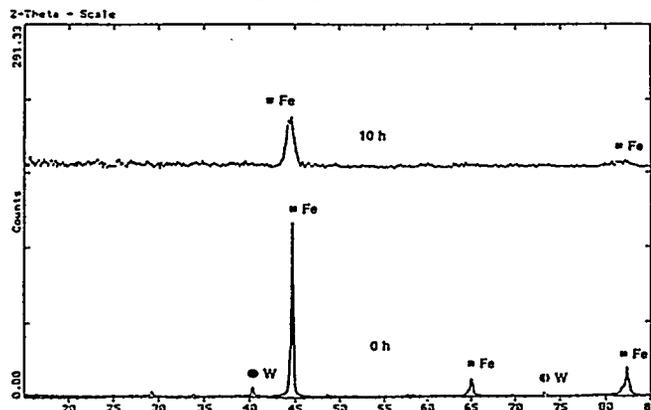


Figure 1: X-ray diffraction patterns of the ODS-13.5Cr powder as a function of milling time.

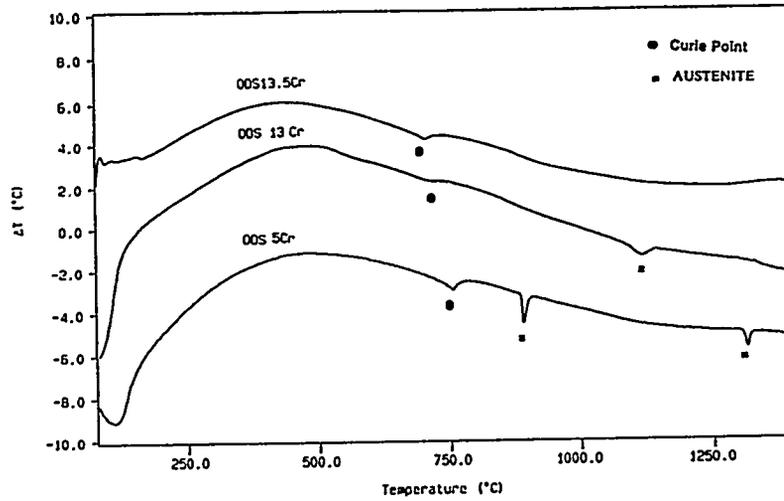


Figure 2: DTA plots of the ODS-5, 13 and 13.5 Cr powders milled for 10 h showing no austenite transformation only in ODS-13.5 Cr.

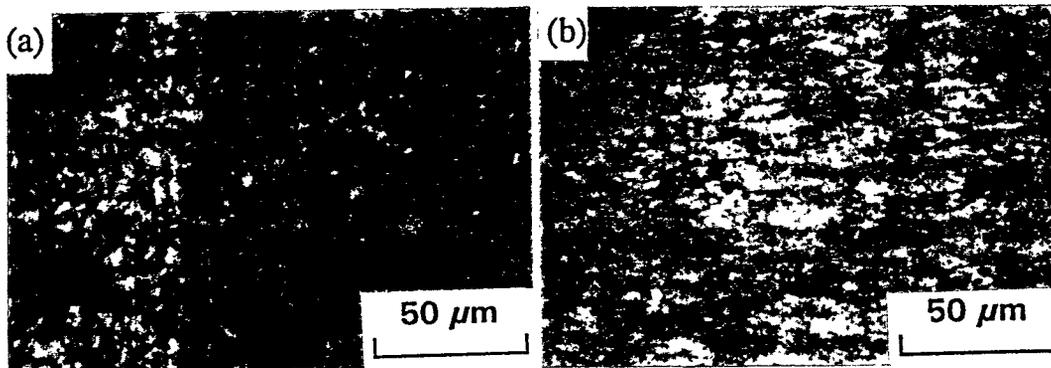


Figure 3: Polished and etched (Vilella's reagent) sections of as-swaged ODS-13.5 Cr material are shown in (a) transverse (b) longitudinal directions.

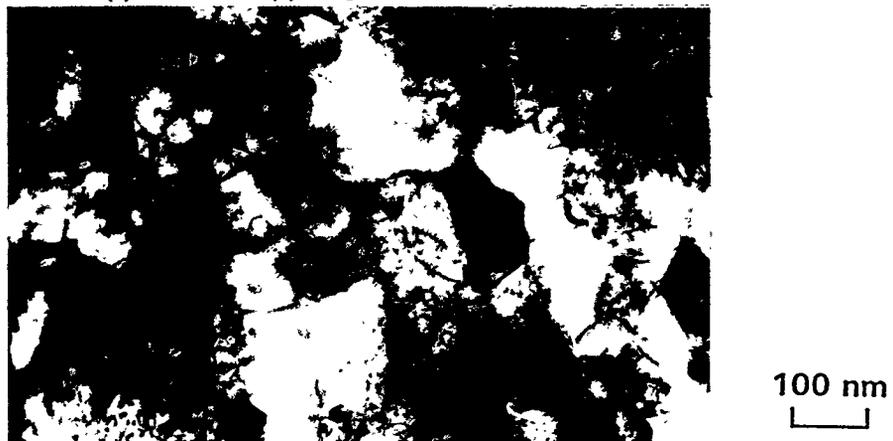


Figure 4: A TEM micrograph showing the elongated grains in the longitudinal direction of the ODS-13.5Cr as-swaged material.

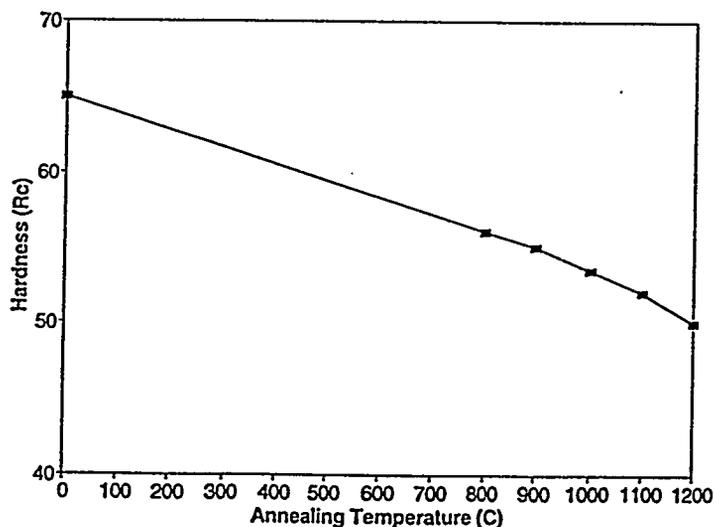


Figure 5: A plot of hardness versus annealing temperature (one hour anneals) indicating a slight decrease in the hardness with temperature.

## CONCLUSIONS

A low-activation grade ODS ferritic steel has been optimized (Fe-13.5Cr-2W-0.5Ti-0.25Y<sub>2</sub>O<sub>3</sub>) and successfully manufactured by mechanical alloying. High material homogeneity was observed by electron microscopic study. Hardness remains very high even following a 1200°C annealing treatment indicating a highly stable microstructure.

## FUTURE WORK

Thermal creep testing of the ODS-13.5Cr alloy is planned for the next reporting period. A transmission electron microscopy study will also be carried out on the crept material.

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## REFERENCES

1. J.J. Fischer, U.S. Patent 4,075,010 (February 21, 1978).
2. D.S. Gelles, *ISIJ Intl.* 30 (1990) 905.
3. D.S. Gelles, DOE/ER-0313/16 (1994) 146.
4. A.N. Niemi, M.G. McKimpson and D.S. Gelles, DOE/ER-0313/6, (1989) 187.
5. A.N. Niemi, M.G. McKimpson and D.S. Gelles, DOE/ER-0313/8, (1990) 177.
6. F.B. Pickering, *Physical Metallurgy and the Design of Steels*, Applied Science Publishers Ltd., Essex, UK, 1978.