Cold compaction of ODS 9Cr martensitic steel powder synthesized by mechanical alloying

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Oxide dispersion strengthened (ODS) steels are advanced class of nanoscale composite materials whose microstructure can be controlled through incorporation of oxide particles by mechanical alloying (MA). This paper deals with the methodologies attempted for cold compaction followed by subsequent sintering of ODS steel of composition Fe-9.2Cr-2W-0.2Ti-0.28V-0.01Mn-0.01Si-0.152C-0.02Ni-0.008Mo-0.33Y₂O₃ synthesized via mechanical alloying of elemental powders. Microstructural studies of the compacted and vacuum sintered specimens after solutionizing heat treatment are reported. As-cold compacted specimens had a green density of 82% TD. Hardness of the heat treated samples is also reported.

Keywords: Oxide dispersion strengthening (ODS), Mechanical alloying (MA), Cold compaction, Energy dispersive analysis of X-rays (EDAX)

Development of low or reduced-activation materials for fusion reactors has its focus on the issue of radioactive waste disposal/recycling of materials from fusion power plant components after they have reached the end of their service lifetime. Hence, the choice of viable candidate materials for structural applications in a nuclear power plant has been narrowed down to (i) ferritic-martensitic steels (ii) SiC/SiC composites and (iii) vanadium alloys. Of these potential candidates, SiC/SiC composites and vanadium alloys are yet to reach the commercialization stage in comparison to steels and require considerable research to reach engineering feasibility. Among the steels, 9 and 12% Cr steels possess improved creep-rupture strengths combined with good oxidation and corrosion resistance at elevated temperatures compared to 2.25%Cr steels. Based on a series of irradiation tests, 8-9% Cr instead of 12%Cr was selected as an optimum level for advanced power plants, since lower Cr content avoids severe hardening and embrittlement problems associated with the formation of fine dispersions of the γ phase. Within the ferritic/martensitic steel class, analogs of commercial steels with Cr, W, V and Ta as alloying elements have been developed with both low activation potential and better radiation resistance than the parent compositions.

Increasing the operating temperature of these alloys to 873 K or 923 K would expand the design window of these alloys, making them more attractive for advanced power systems. However, solid solution strengthening results in alloys with limited ductility, while precipitation strengthening results in coarsening of precipitates at higher temperatures. To overcome these limitations, Oxide dispersion strengthening (ODS) of the alloys with ultra fine oxide particles by mechanical alloying (MA) appears to be the most feasible route to produce technologically superior materials for applications at higher temperatures. The present study aims at the consolidation of ODS 9Cr martensitic steel using elemental powders prepared via MA by cold compaction followed by sintering. Microstructural characterizations of the compacted specimens are reported.

Experimental Procedure

Synthesis of ODS alloy powders

ODS alloy powders were synthesized using a double cone blender by MA of elemental metal powders to arrive at the following chemical composition (in wt%): Fe-9.2Cr-2W-0.2Ti-0.28V-0.01Mn-0.01Si-0.152C-0.02Ni-0.008Mo-0.33Y₂O₃. The ball to charge ratio was maintained as 10:1 and 1 wt% stearic acid was used as the process control agent. Steel bearing balls of 6 mm diameter were

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used as the grinding media. The blender was operated at 40 rpm and the steel powder charge was blended for a duration of 25 h. The XRD analysis of the milled powders showed that all the individual constituents have gone into solution (Fig. 1). Detailed characterization studies of the synthesized steel powders and the subsequent optimization of the milling parameters have been reported elsewhere

Cold pressing of ODS alloy powders

The mechanically alloyed powders were thoroughly mixed with 0.6 wt% of lubricant containing 2.1% Zn for achieving good green strength. MA powders along with lubricants were blended for 40 min at 20 rpm. The blended powders were cold compacted employing a DAEGHA (Korean make) press into specimens which had 82% TD along with good surface finish (Fig. 2).

One of the cold compacted specimens was then vacuum sintered at 10^{-5} Torr vacuum for 210 min at 1273 K. Three cold compacted samples were sintered at 1073 K for 180 min. After sintering of the three specimens, one each was subjected to furnace cooling, air cooling and quenching respectively. Microstructural characterizations of the three specimens were carried out employing a Nikon optical microscope and JEOL JSM 6360 scanning electron microscope (SEM). Microhardness measurements of the cold compacted, sintered, solution treated and heat treated samples were carried out employing a Mitutyo microhardness tester with diamond indenter using a load of 50 g.

Results and Discussion

Characterization of vacuum sintered specimens

The optical micrographs of vacuum sintered ODS steel specimens indicate the formation of a two-phase microstructure with a uniform distribution of the second phase in the matrix (ferrite). Figure 3 depict the unetched and etched microstructures of the vacuum sintered ODS steel specimens respectively.

The SEM/EDAX analysis indicated a substantially uniform two-phase microstructure with significantly lower fraction of second phase containing slightly higher chromium content. (Table 1) Area analysis employing EDAX confirmed the presence of the various alloying elements of the ODS steel (Fig. 4).

Characterization of sintered and furnace cooled specimens

The EDAX results of the cold compacted, sintered and furnace cooled specimen is given in Table 2. Typical elemental X-ray distribution images of the same specimen showing the presence of yttrium along with tungsten are shown in Fig. 5.

Characterization of sintered and air cooled specimen

The SEM examination of the sintered and air cooled specimen indicated the formation of a two-phase microstructure. Area analysis by EDAX gave the composition of the individual alloying elements as shown in Fig. 6 and it can be seen that in spite of the two phase structure the concentration of the various elements reasonably correspond to the starting composition. X-ray distribution images indicated the

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<th>Sl. No</th>
<th>Analyzed Region</th>
<th>wt %</th>
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<tr>
<td></td>
<td>Fe</td>
<td>Cr</td>
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<tr>
<td>1</td>
<td>Area (Matrix)</td>
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<td>2</td>
<td>Spot (2nd phase)</td>
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Fig. 4—Area analysis employing EDAX, confirms the presence of all elements

Table 2—Combined EDAX results of the sintered and furnace cooled specimen

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<th>Analyzed region</th>
<th>wt %</th>
<th>Fe</th>
<th>Cr</th>
<th>W</th>
<th>Y</th>
<th>Ti</th>
<th>V</th>
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<td>1.78</td>
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Table 3—Combined EDAX results of sintered and air cooled specimen

<table>
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<tr>
<th>Analyzed Region</th>
<th>wt %</th>
<th>Fe</th>
<th>Cr</th>
<th>W</th>
<th>Ti</th>
<th>V</th>
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<td>8.19</td>
<td>2.41</td>
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<td>-</td>
<td>0.28</td>
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Fig. 5—X-ray images of sintered and furnace cooled specimen
Characterization of sintered and quenched specimen

The SEM/EDAX analysis of the cold compacted and quenched specimen shown in Fig. 8 indicates the desired starting composition of the alloying elements after consolidation. Figure 9 represents the X-ray distribution images of the same specimen wherein, the presence of yttrium along with tungsten is evident. The results show that the area analysis gives concentrations of various elements corresponding to the starting composition.
Preliminary mechanical property evaluation of ODS steel specimens

The microhardness measurements recorded for the cold compacted, vacuum sintered specimen and cold compacted heat treated specimens are given in Table 4. Hardness values indicate that quenched specimen shows the highest value while furnace...
cooled specimen shows the lowest value which can be attributed to the relatively faster cooling rates in quenching as compared to annealing.

Conclusions

The following conclusions can be drawn from this study:

ODS 9Cr martensitic steel successfully prepared by mechanical alloying of elemental powders has been cold compacted and sintered in vacuum.

The compacts have been sintered at 1073 K for 3 h and were subjected to furnace cooling, air cooling and quenching.

The SEM/EDAX analysis of the above samples showed two-phase microstructure, while the sintered and air cooled and quenched specimens gave the best elemental distribution corresponding to the starting composition.

Microhardness measurements show that the quenched specimen records highest hardness.

Acknowledgement

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References