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Effect of Yttrium Oxide Dispersion on the Microstructure and Properties of Tungsten Heavy Alloys

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ABSTRACT

Tungsten heavy alloys are considered as two phase composites with 88 to 97 wt% tungsten interspersed in a matrix of relatively low melting elements such as nickel, iron and cobalt. The mechanical properties of these alloys are greatly influenced by the microstructural features such as tungsten grain size, tungsten-tungsten contiguity and matrix volume fraction. Oxide dispersion strengthening (ODS), refinement of tungsten grain size, cyclic heat treatment, addition of alloying elements like Cr, Mo, and Co are some of the methods investigated to improve the microstructural features and thereby the mechanical properties of tungsten heavy alloys. Among these methods ODS has been considered as a promising processing technique since the tungsten grain size observed in ODS alloys is finer compared to the conventional alloys and more importantly the dynamic fracture mode changes from adiabatic shear band to brittle fracture. The present study is mainly focused on investigating the effect of 0.3 wt% yttrium oxide (Y_2O_3) dispersion on the microstructure and consequently the tensile properties of 90W-6Ni-2Fe-2Co alloy. With 0.3 wt%Y_2O_3, the ODS alloy (89.7W-6Ni-2Fe-2Co-0.3Y_2O_3) is processed by two-stage sintering with subsequent thermo-mechanical treatment which includes vacuum heat treatment and swaging. ODS alloy and the conventional alloy (without oxides) are compared based on the microstructures and tensile properties obtained after liquid phase sintering and after final processing.

Keywords: Oxide dispersion; Yttrium oxide; Two stage sintering; Fine tungsten grains

1. INTRODUCTION

Mechanical properties of tungsten heavy alloys (WHAs) are influenced by their microstructural features such as tungsten (W) grain size, matrix (M) volume fraction and W-W contiguity[1]. Several investigations have explored the possibility of improving the mechanical properties by alloying addition, mechanical alloying and cyclic heat treatment[2-6]. Among these methods, oxide dispersion strengthening gained significant importance because of its potential in refining the W grain size[6]. Ryu[6], *et al.* have reported that ODS alloys have a tendency for brittle fracture rather than a localised deformation during high strain rate shear deformation. Oxides present at the W-M interfaces result in W-W interfacial debonding. Hence, the fracture behaviour of these ODS alloys has been studied extensively to explore its advantages.

ODS involves the use of fine oxide dispersions in the alloy to achieve better mechanical properties. Thermodynamically stable fine oxides like yttrium oxide (Y_2O_3) are dispersed in the matrix of a tungsten heavy alloy. Based on the oxide content, this method is advantageous in attaining high temperature strength[7]. Y_2O_3 could be present at the W-M interface and also within the tungsten grains and inhibits the growth of the tungsten grains. Hong[7], *et al.* have reported refinement of tungsten grain size by increasing the oxide content from 0.1 wt% to 5 wt%. The amount (in weight %) of yttrium oxide dispersed in an alloy has a significant influence on its microstructure and mechanical properties[7]. Y_2O_3 less than 0.1 wt% does not contribute in refinement of grain size whereas above 5 wt%, tensile properties deteriorate[7]. If the oxide content is high, the oxides get inhomogeneously distributed at the W-M and W-W interfaces. These oxides then act as fracture initiation sites during mechanical testing and result in inferior tensile properties by means of brittle fracture[7]. In the present study, the amount of oxide content and the processing parameters have been optimised based on the observations reported by Lee[8], *et al.*

Very similar to conventional alloys, ODS alloys are also processed using higher sintering temperatures and longer soaking times to achieve full densification. However, this practice leads to the formation of coarse tungsten grains. In order to avoid grain coarsening, a two-stage sintering cycle followed by heat treatment has been reported by Lee[8], *et al.* to have a better control over the microstructure of ODS alloys. In two-stage sintering, solid state sintering (SSS) is carried out between 1300 °C and 1450 °C for 1 h - 2 h and then subsequently liquid phase sintering (LPS) between 1465 °C and 1485 °C for 1 h. Solid state sintering results in near full

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density of the alloy while tungsten grain growth and contiguity can be controlled by using lesser holding time in the liquid phase sintering stage[8]. Densification upto 97 per cent can be achieved after solid state sintering but the microstructure will have contiguous tungsten grains and isolated matrix pools. With subsequent liquid phase sintering, contiguous tungsten grains change to spherical tungsten grains that are uniformly embedded in the matrix[8]. Finer tungsten grain size has been reported in the two-stage sintered ODS alloys compared to conventional alloys[8].

2. PRESENT STUDY

The present study is aimed at understanding the effect of 0.3 wt% yttrium oxide dispersion on the microstructure and mechanical properties of 90W-6Ni-2Fe-2Co alloy. With 0.3 wt% yttrium oxide, the ODS alloy obtained is 89.7W-6Ni-2Fe-2Co-0.3Y_2O_3. ODS alloy is processed by using two-stage sintering with subsequent heat treatment and swaging. Microstructural characterisation and evaluation of tensile properties are carried out after every stage of processing. A comparison has been drawn between the conventional alloy and the ODS alloy based on the microstructure and the properties obtained after liquid phase sintering and after final processing (second stage thermo-mechanical treatment).

3. EXPERIMENTAL

Tungsten, nickel, iron and cobalt powders of commercial grade were used in the present study to obtain 90W-6Ni-2Fe-2Co and 89.7W-6Ni-2Fe-2Co-0.3Y_2O_3 alloys. Details of tungsten, nickel, iron and cobalt powders are listed in Table 1. Particle morphology has been studied using scanning electron microscopy (SEM) and is as shown in Fig. 1(a) to 1(e). Yttrium oxide powder has an average particle size of 3µ and it is mixed with tungsten, nickel, iron and cobalt powders in a conventional ball mill for 24 h using stainless steel balls with 2:1 BPR (Ball to Powder Ratio). Subsequently, the powder mixture was reduced in hydrogen atmosphere at 700 °C for 1 h. The powder mix is then cold isostatically pressed (Make: National Forge, Belgium) at 200 MPa into green compacts of dimensions 40 mm diameter x 200 mm height. Two stage sintering was carried out in a pusher type FHD furnace (Make: FHD Furnaces Ltd, England, Max.Temp. 1800 °C). Solid state sintering at 1300 °C for 2 h and liquid phase sintering at 1470 °C for 1 h were carried out under hydrogen atmosphere. First stage vacuum heat treatment at 1150 °C for 4 h (vacuum level should be minimum 10^{-4} mbar) with subsequent oil quenching was carried out after the two stage sintering. Heat treated alloy was then subjected to first stage swaging from 38 mm to 34 mm diameter (deformation of 20 %) using a rotary swaging machine. Subsequently, swaged alloys were subjected to second stage vacuum heat treatment at 1150 °C for 4 h. Second stage swaging was carried out from 34 mm to 30 mm diameter which resulted in a deformation of 22 per cent. Both conventional process and oxide dispersion strengthening are as shown in Fig. 2. Conventional liquid phase sintering is also carried out in the same pusher type FHD furnace. Unlike ODS alloy, conventional alloy is subjected to single stage sintering at 1460 °C for 2 h. Rest of the processing which includes

Table 1. Properties of tungsten, nickel and iron powders (as-received grade)

Parameters	Tungsten	Nickel	Iron	Cobalt
Apparent density (g/cc)	4.7 ± 0.2	2.1 ± 0.1	3 ± 0.2	3.1 ± 0.2
Tap density (g/cc)	7.1 ± 0.2	4.2 ± 0.1	4.2 ± 0.1	4.4 ± 0.2
Particle shape	Cuboidal	Irregular	Spherical	Irregular
Median size (D_{50}) x10^{-6} m	26.1 ± 1.2	27.1 ± 1.1	7.2 ± 0.9	18 ± 1.2

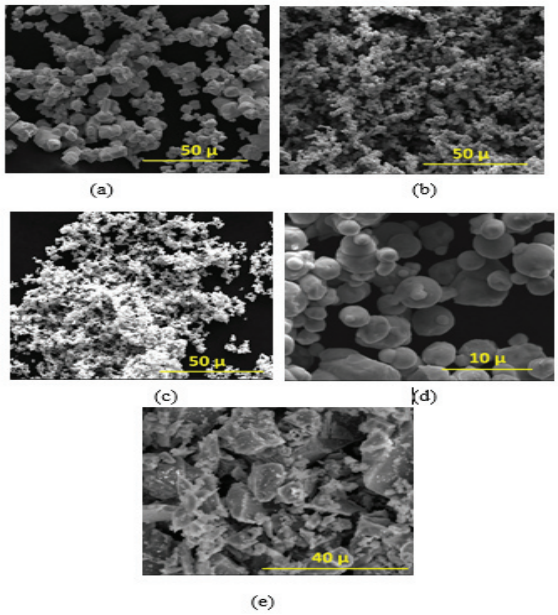

Figure 1. SEM images of as-received: (a) tungsten powder, (b) nickel powder, (c) cobalt powder, (d) iron powder, and (e) yttrium oxide.

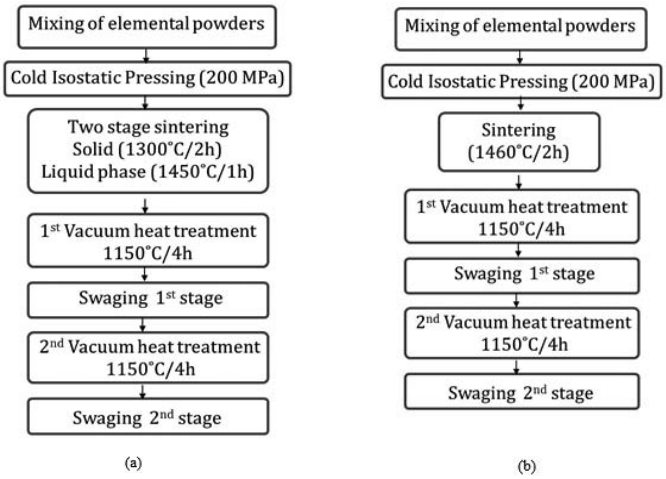

Figure 2. Comparison between (a) oxide dispersion process and (b) conventional process.

vacuum heat treatment and swaging is maintained similar to that of ODS alloy.

Microstructure of ODS alloy was studied in solid state sintered (SSS) stage, liquid phase sintered (LPS) stage and after first and second swaging. Samples for microstructural evaluation were prepared by standard metallographic procedures and

were observed under scanning electron microscope (SEM: Make: FEI Quanta 400 ESEM). Microstructural characterisation included determination of grain size, contiguity and dihedral angle. Grain size and dihedral angle measurements were made using Image analysis (Make: Image J). Contiguity was calculated using the line intercept method and the formula

$$\frac{2Nww}{(2Nww + Nwm)} \quad (1)$$

wherein N_{WW} is the number of W-W contacts and N_{WM} is the number of W-M contacts determined by placing a grid the microstructural image and counting the contact points. 100 measurements were made using this method to determine contiguity. Tensile properties were evaluated using a tensile testing machine (Model: Instron 5500R Universal tensile testing machine). Tensile fractographs were also studied using scanning electron microscope.

Table 2. Comparison of microstructural features between ODS alloy and conventional alloy in different processing stages

Processing steps	Contiguity		Dihedral angle (deg.)		W grain size (μ)	
	ODS alloy	Conventional alloy	ODS alloy	Conventional alloy	ODS alloy	Conventional alloy
Liquid phase sintering	0.35 ± 0.13	0.62 ± 0.2	55 ± 18	62 ± 21	32 ± 6	42 ± 18
First heat treatment and swaging	0.23 ± 0.08	--	32 ± 20	---	26 ± 10	---
Second heat treatment and swaging	0.20 ± 0.1	0.4 ± 0.1	28 ± 19	32 ± 6	16 ± 7	34 ± 5

4. RESULTS

Microstructures of 89.7W-6Ni-2Fe-2Co-0.3Y_2O_3 alloy obtained after SSS and LPS stages are as shown in Figs. 3(a) and 3(b). Highly contiguous, non-spherical tungsten grains are observed in SSS stage whereas after LPS stage, matrix penetration between tungsten grains has changed the grains to rounded tungsten grain. Average grain size obtained after two stage sintering is listed in Table 2. Microstructure of conventional alloy after LPS stage is shown in Fig. 3(c). ODS alloy has finer tungsten grains compared to the conventional alloy (as listed in Table 2). Microstructures of ODS alloy obtained after first and second stage thermo-mechanical treatments are as shown in Figs. 4(a) and 4(b). Grain size of ODS alloy, as listed in Table 2, is found to decrease after every step of thermo-mechanical treatment.

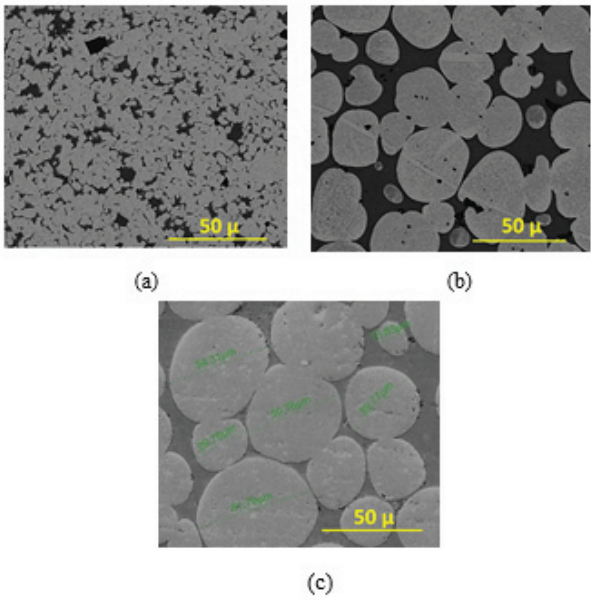

Figure 3. SEM microstructures of ODS alloy: (a) solid state sintered, (b) liquid phase sintered, and (c) liquid phase sintered conventional alloy.

Line intercept method, which is used for calculating contiguity is as shown in Fig. 5(a) and the contiguity values of both conventional and ODS alloys are listed in Table 2. Dihedral angle measurements carried out using image analysis are shown in Fig. 5(b) and the values are listed in Table 2. As listed in Table 2, tungsten grain size, contiguity and dihedral angle are found to be higher in the conventional alloy in both LPS stage and after second swaging.

Tensile properties of ODS alloy have been evaluated after both SSS and LPS stages of sintering and also after every stage of thermo-mechanical treatment. Tensile properties are listed in

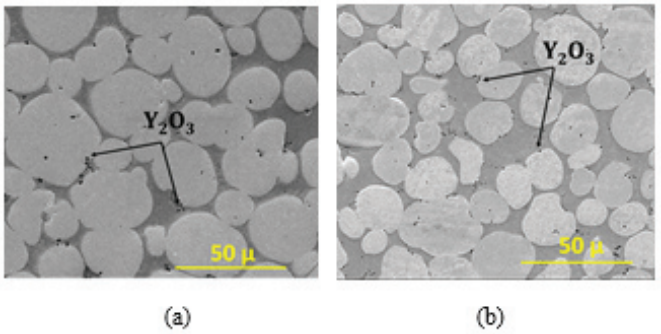

Figure 4. SEM microstructures of ODS alloy: (a) first stage heat treated and swaged and (b) second stage heat treated and swaged.

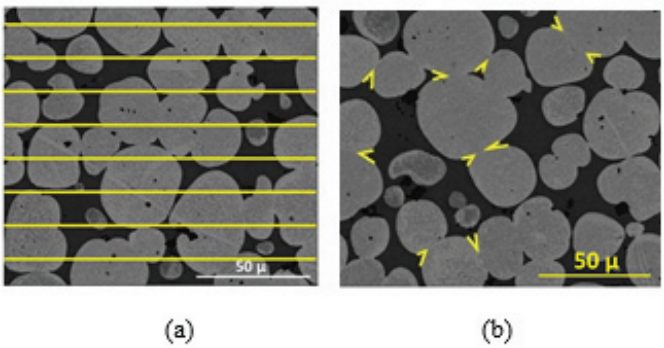

Figure 5. Indicative figures: (a) line intercept method of calculating contiguity and (b) dihedral angle measurements.

Table 3. Among all the stages, solid state sintered (SSS) alloy has the most inferior combination of properties. However, tensile properties improve after liquid phase sintering and also after two stages of thermo-mechanical treatment. Comparison of tensile properties ODS alloy and conventional alloy is listed in Table 3. This comparison is made in the LPS condition and after second swaging (final processing); very similar to the comparison made between microstructural features. It is observed that the properties of ODS alloy are marginally inferior to the conventional alloy.

Table 3. Comparison of tensile properties between ODS alloy and conventional alloy in different processing stages

Processing step	ODS alloys		Conventional alloys	
	UTS (MPa)	Elongation (per cent)	UTS (MPa)	Elongation (per cent)
Solid state sintering	545	4	-	-
Liquid phase sintering	686	5	682	4
After final processing (second swaging)	1311	8	1400	10

Fractographs of tensile tested samples are shown in Figs. 6(a) and 6(b). Contiguous tungsten grains without any indication of matrix failure is observed in the fractograph of SSS stage (as shown in Fig. 6(a)). In the LPS stage, both W-W debonding and W-M interface failure are observed in the tensile fractograph (Fig. 6(b)). Figures 6(c) and 6(d) show the tensile fractographs of first and second stage thermo-mechanically treated samples. Evidences of both tungsten grain cleavage and matrix rupture are observed after first stage heat treatment and swaging whereas second stage sample exhibits predominant tungsten grain cleavage.

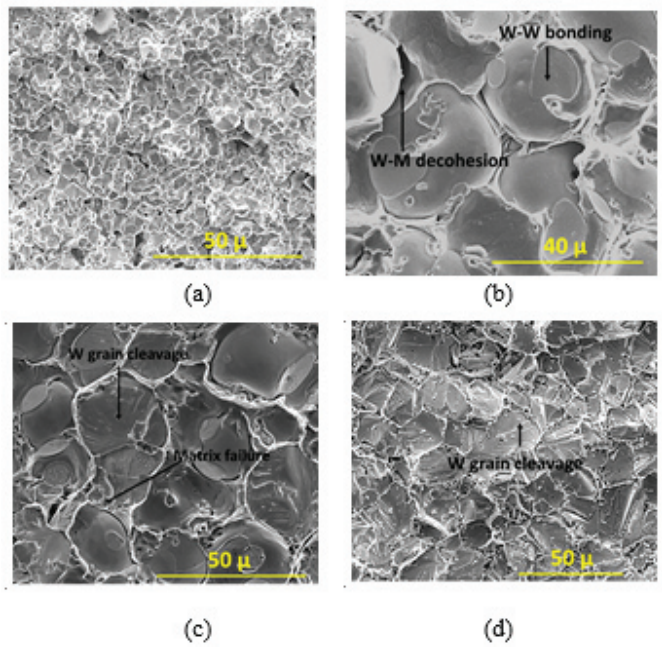

Figure 6. Tensile fractographs of ODS alloy: (a) solid state sintered, (b) liquid phase sintered, (c) first stage heat treated and swaged, and (d) second stage heat treated and swaged.

5. DISCUSSIONS

In the case of conventional alloy (without oxides), after LPS, the presence of very coarse tungsten grains (as indicated in Table 2) substantiates the fact that tungsten grains have experienced substantial amount of grain coarsening. This grain coarsening during conventional LPS occurs by Ostwald ripening mechanism in which smaller tungsten grains dissolve in the matrix and reprecipitate on larger grains[9]. However, on the contrary, tungsten grain coarsening is not significant in ODS alloys as grain growth inhibition and retardation of grain coarsening occurs because of the presence of insoluble oxides. Ryu[6], *et al.* reported that the grain size decreases as oxide content is increased upto 5 wt% and these oxide dispersoids located at the W-M interfaces block the diffusion flow between the tungsten and the matrix[6]. It has also been reported that the diffusion distance for solute atoms increases with the presence of second phase particles[10]. In the present study, grain size control can therefore be attributed to the presence of yttrium oxides in the W-M interface. Similar observations of grain size reduction wherein oxide dispersed alloy has an average tungsten grain size of 15 µ against 27 µ of a base alloy has been reported in 93W-4.9Ni-2.1Fe alloy with 0.1 wt % oxide content[11]. In the present study, as listed in Table 2, average grain size is 32±6µ after LPS and 16±7µ after second swaging. Apart from tungsten grain size, another major microstructural factor that influences the mechanical properties of tungsten heavy alloys is the W-W grain contiguity.

Average fraction of a tungsten grain that is in contact with other solid tungsten grains is referred to as W-W contiguity[12]. In the SSS condition since the microstructure is not completely developed and there are no clear W-W and W-M interfaces, hence, it is not possible to calculate contiguity. However, in the LPS condition, the interfaces become prominent because of the matrix penetration between tungsten grains and hence the contiguity value can be calculated (indicated in Table 2). Contiguity calculations made using line intercept method can be validated using the dihedral angles determined using image analysis. Angle at the triple point junction between two solid grains and the matrix is referred to as dihedral angle. Contiguous grains which are not separated by the matrix have high dihedral angle between them. Similar observations have been made in the present study; dihedral angle decreases with decreasing contiguity (Table 2).

In the solid-state sintered ODS alloy, effect of W-W contiguity is prominent as the alloy exhibits poor tensile strength and elongation (Table 3) understandably because of the presence of contiguous tungsten grains. However, in the liquid phase sintered condition (Table 3), the tensile properties improve, as the matrix penetrates between the tungsten grains, resulting in decrease of W-W contiguity and increase in the matrix volume fraction. In the LPS condition, ODS alloy has tensile properties that are similar to those of conventional alloy, but as discussed earlier ODS alloy exhibits finer tungsten grains compared to the conventional alloy.

In order to increase the tensile strength and elongation, heat treatment is introduced after LPS. Heat treatment of heavy alloys in vacuum results in the following advantages:

(i) removal of dissolved hydrogen from the matrix and

(ii) increase of homogeneity in the alloy chemical composition[13].

In the present study, vacuum heat treatment is carried out at 1150 °C for 4 h with subsequent oil quenching. Heat treated alloys are then subjected to deformation by swaging. Swaging results in improvement in tensile strength and reduction in ductility. So, a combination of heat treatment and swaging is carried out to obtain optimum tensile strength and ductility.

Katavic[13], *et al*. have reported that hardening observed during the initial 15 per cent to 20 per cent deformation is the result of hardening of both the tungsten and the matrix phase. On the other hand, with increasing deformation, tungsten hardening becomes dominant while matrix phase hardening reduces. Also, increasing the amount of deformation results in increase in tensile strength and decrease in elongation to failure[14]. In the present study, first stage swaging with 20 per cent deformation (38 mm to 34mm) results in the increase of tensile strength from 686 MPa to 788 MPa and elongation from 5 per cent to 8 per cent. Subsequent second stage heat treatment and swaging with 22 per cent deformation (34 mm to 30 mm) results in the increase of tensile strength to 1311 MPa while the elongation remains at 8 per cent. In the present study, with increasing deformation, from first to second stage swaging, contiguity has decreased marginally; tensile strength has increased substantially while elongation to failure remained unaffected. Strain hardening induced by first swaging results in increase in tensile strength from 686 MPa to 788 MPa. Though second heat treatment is expected to decrease the strength level marginally, second swaging results in increase of tensile strength to 1311 MPa.

In tungsten heavy alloys, crack initiation is usually at the weakest interface i.e., the W-W interface but crack propagation depends on the strength of the other interface, namely the W-M interface. Contiguous tungsten grains result in poor tensile properties (Table 3) since the W-W grain interfaces, being the weakest links, fail even at relatively minor loads.

In the LPS condition, the tensile fractograph shows both W-W debonding and W-M interface failure (Fig. 6(b). The W-M interface also being a weaker interface (in the sintered condition) provides low energy crack path. Both the W-W debonding and W-M interface failure leave the W grains intact. This type of failure is a typical intergranular failure which is usually not associated with superior tensile properties. In present study, both tensile strength and elongation have improved (Table 3) between SSS and LPS conditions. In order to improve the properties further, liquid phase sintered alloys are subjected to thermo-mechanical treatments.

After first stage heat treatment and swaging, fracture surface of tensile tested sample presents evidence of W grain cleavage and matrix rupture. Observation of W grain cleavage, though not predominantly, indicates that the crack has propagated through the grain. This implies the presence of strong W-M interface. Increase in tensile strength from 686 MPa to 788 MPa can be ascribed to tungsten grain cleavage. Ductile failure of the matrix results in the increase of elongation from 5 per cent to 8 per cent. However, in the second stage sample, tungsten grain cleavage is clearly a predominant feature, which explains the substantial increase in tensile strength from 788 MPa to 1311 MPa, whereas the unchanged elongation (8 per cent in both the first and second swaged condition) can possibly be ascribed to a balance between strain hardening and decreasing contiguity. Thus, the fractographic features are found to be consistent with the tensile properties obtained after two stages of sintering and two stages of thermo-mechanical treatments.

Though tensile properties of ODS alloys are not on par with conventional alloys, the advantage of fine tungsten grains and cleavage fracture of tungsten grains, is reported to be beneficial in achieving the desired fracture mode. Moreover, there is a scope to enhance the properties of the alloy by altering the base composition and also thermo-mechanical treatment.

6. CONCLUSIONS

(i) ODS alloy is prepared by addition of 0.3 wt% yttrium oxide to conventional 90W-6Ni-2Fe-2Co alloy. The resultant 89.7W-6Ni-2Fe-2Co-0.3Y_2O_3 alloy is processed using two stage sintering, unlike single stage conventional liquid phase sintering.

(ii) A comparison after sintering indicates that the ODS alloy has finer tungsten grain size and tensile properties similar to conventional alloys. Two-stage sintering along with the presence of oxides along the interfaces has resulted in lesser grain coarsening.

(iii) Two-stage sintered ODS alloy is subjected to successive thermo-mechanical treatments (heat treatment followed by swaging) in two stages; first and second. Tensile strength has increased in each stage whereas elongation remained unaffected between the two stages. The ODS alloy has marginally lower tensile properties but finer tungsten grains as compared to the conventional alloy.

(iv) Though oxide dispersion is aimed at achieving finer grain size and brittle fracture during dynamic deformation, the amount of oxides and the uniform dispersion of oxides play a vital role in determining the properties. In the present study, the marginal decrease in tensile properties could possibly be because of the amount and non-uniform distribution of oxides along the interfaces.

(v) The effect of second heat treatment and swaging on grain refinement is yet to be ascertained and shall be reported in due course with appropriate analysis using TEM.

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In the current study, he is involved in processing of ODS alloys in DMRL. Processing includes mixing, compaction, sintering and heat treatment stages. Characterisation of microstructure and evaluation of mechanical properties.

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In the current study, he has contributed immensely and guided in replying to the reviewers queries. Contributed in detailed characterisation of the alloys including fractography and image analysis.

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In the current study, he has initiated the concept and guided in experimental work. Reviewed the manuscript and incorporated several suggestions.