Development of 410S-Y2O3 oxide dispersion strengthened steel prepared by a powder metallurgy technique

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Abstract. In the present study, the Oxide Dispersion Strengthened (ODS) steels were synthesized by adding various concentration of Y2O3 (0, 0.25, 0.35 and 1 wt. %) into 410S steel. The sample preparation was carried out using a powder metallurgy technique. The steel-Y2O3 powders were wet mechanically alloyed for 3 h, dried and then compacted into green body. The samples were sintered in a vacuum furnace at elevated temperatures of 1000 °C and 1100 °C for 3 h. The phase analysis and structural characterization were carried out by XRD and SEM-EDX, respectively. The bulk density and hardness of the samples were measured using Archimedes principle and Vickers hardness tester, respectively. According to the results of metallographic characterization, ODS steels are composed mainly by (Fe,Cr) phase. The SEM observations indicate the presence of precipitates in the sintered steels. The density and hardness of the sample sintered at 1000 and 1100 °C increase with the increase of Y2O3 concentration. At 1100 °C, however, the density and hardness of ODS steel containing 1 wt. % Y2O3 have a tendency to decrease due to the formation of a compound consisting of Fe, Cr, Mn, Si and O.

1. Introduction
High temperature applications such as advanced coal fired power plant (A-USC) and nuclear reactor require material that are resistant not only to oxidation and corrosion but also to mechanical loading. Moreover, for nuclear energy systems it must withstand to high radiation damage, swelling and embrittlement [1, 2]. Currently, oxide dispersion strengthened (ODS) steel is the most promising candidate material for the aforesaid applications due to the fact that it exhibits an excellent high temperature properties over the conventional heat resistant steels without oxide particle dispersoids [3]. Structurally, the fine oxide particles are dispersed uniformly in the matrix. Their presence can improve the creep resistance, mechanical strength and irradiation resistance [4, 5].

Mostly, the ODS steel is based on Y2O3 fine particles which was added into Fe-Cr [5-6] and Fe-Ni based powder [7]. The particles were acted as a stabilizer of free dislocation and sub-grain structure [8]. Consequently, their structure, size and distribution become important factors that determine the ODS properties. The finer dispersoids have a significant effect on the hardness and strength of ODS material. Previous study reported that the addition of Ti to Y2O3 strengthened ODS steel can refine the Y-Ti-O oxide particles [3, 4] into 2-6 nm and increases the number density of oxide particles in the
matrix. In comparison to the $Y_2O_3$-ODS steel without Ti addition, $Y$-Ti-O strengthened ODS steel exhibits a better creep and radiation resistance, and also higher elevated temperature strength [2].

Several techniques such as spark plasma sintering have been used to synthesize the ODS steel. However, the issues of anisotropic properties and production cost of this technique are often taken into consideration in its use [5]. On the contrary, due to its cost effectiveness and uniqueness, conventional powder metallurgy technique offers distinctive benefit for fabrication of high temperature materials. This method usually consisted of subsequent process as powder blending, addition of pressing aids, compaction, sintering and further thermal treatment to obtain the desired microstructure and properties. Mostly, a mechanical alloying technique was used to homogenize metallic powders and disperse the fine ceramic powders as $Y_2O_3$ to the matrix [3-5, 8]. The small concentration of sintering aids or binder can be added to obtain the high strength of green body. The powder compaction or consolidation was performed at room temperature by applying compressive stress to the powders. This results a green body with a porosity of about 10-60%. In order to get high end of final density, the compact was sintered at elevated temperature in a carefully controlled atmosphere [9]. The aforementioned points suggest that the material properties of final product are strongly depending on the processing parameter used.

In the present study, the fabrication of ODS steels was investigated by adding the varying concentration of $Y_2O_3$ into steel 410S. This material was considered as starting material because it contains Ti element which may play a role in the formation of fine dispersed oxide particles. The structures of ODS steels after sintering at 1000 and 1100 °C were studied by means of XRD and SEM-EDX. In this paper, the density and hardness of ODS steels were also presented.

2. Experimental method

2.1. Sample preparation

The ODS steels powder containing varying amount of $Y_2O_3$ were prepared using a conventional sintering method. First, the steel powder starting material was weighted according to the stainless steel powder 410S composition, as shown in Table 1.

| Table 1. Chemical element of steel 410S. |
|------------------|---|---|---|---|---|
| **Element**      | **Fe** | **Cr** | **Mn** | **Ti** | **Si** |
| Weight percentage (wt.%) | Balance | 14 | 1 | 0.2 | 1 |

The steel powder was then added with varying concentration of 0, 0.25, 0.35 and 1 wt. % $Y_2O_3$ powders with the size of about 5-10 μm. Each composition was mechanically alloyed in steel vial under wet milling condition with balls and powder ratio of 10 to 1 using a high speed shaker mill at oscillation frequency of 700 rpm/min for 3 h. After milling, the mixture powder was placed in desiccators and dried at ambient temperature and environment. The mechanically alloyed powder was then packed in the steel dies and then compacted under a compressive stress of 8 Ton for 3 min. The samples were then sintered in vacuum atmosphere of 10 Pa at elevated temperature of 1000 °C and 1100 °C for 2 h. Finally, it was cooled down in the same chamber until the furnace reached a room temperature.

2.2. Sample characterization

For metallographic characterization and properties measurement, the surface of sintered steels was ground to SiC abrasive papers from #800 to #2000 and DP-polishing cloth for up to mirror finish. The phase composition of ODS powders before and after sintering was determined by X-ray diffractometer (XRD Smartlab Rigaku). The XRD scanning was performed using a Cu Kα irradiation (40kV, 30mA) at diffraction angle of 10-90 deg, step width of 0.01 deg, and scan speed of 0.25. The microstructures of the steels were characterized by a scanning electron microscope (SEM Hitachi SU3500) attached
with energy dispersive X-ray spectrometer (EDX Horiba). The sample density was evaluated according to the Archimedes principle. The hardness was measured using Vickers microhardness tester (Leco TM-100 Microhardness) with an applied load of 13 kgf for 28 sec.

3. Results and discussion

3.1. Morphology of the powder after milling

Figure 1 shows the XRD profiles of ODS powders with different amount of Y$_2$O$_3$ content after milling for 3 h.

![X-ray diffraction patterns of ODS powders containing 0, 0.25, 0.35 and 1 wt.% Y$_2$O$_3$ after milling for 3 h.](image)

It can be seen that there is no significant difference in XRD patterns of ODS powders containing varying amount of Y$_2$O$_3$. The graph clearly shows that all powders after milling for 3 h have low diffraction intensity and considerable line broadening. This is attributed to the grain refinement or the formation of nanosize structure. Mainly, the diffraction peaks are found at the diffraction angle of about 44° and 82°. According to the results of XRD analysis, the peaks are detected as (Fe,Cr) phase [3]. It was notable that atomic radius of Fe is almost equal to the Cr. Moreover, they also have BCC crystal structure [10]. Accordingly, the diffraction peaks of both elements are overlap between each other and preferentially form (Fe,Cr) solid solution. The results of XRD characterization also reveal that the XRD analysis didn’t detect the Y$_2$O$_3$ reflection. During mechanical alloying process, the Y$_2$O$_3$ and steel powders were subjected to high energy ball impacts. Thus, the brittle Y$_2$O$_3$ powders are likely to fragment into fine particles. The particles were deeply incorporated or dissolved in the steel matrix during MA [3-4, 11]. Accordingly, their X-ray diffractions become very weak compared to the (Fe,Cr) diffraction peaks.

3.2. Structure of ODS steel after sintering at 1000 °C

Figure 2 shows XRD profiles of ODS steels with various content of Y$_2$O$_3$ after sintering at 1000 °C for 2 h.
Figure 2. X-ray diffraction patterns of ODS steels containing 0, 0.25, 0.35 and 1 wt.% Y₂O₃ after sintering at 1000 °C for 2 h.

Figure 3. BSE Comp images of ODS steels containing (a) 0, (b) 0.25, (c) 0.35 and (d) 1 wt.% Y₂O₃ after sintering at 1000 °C for 2 h.
It is apparent that the diffraction peak of ODS steel after sintering at 1000 °C for 2 h become sharp and stronger compared to the powder after milling. This could be due to the improvement of crystallinity and grain growth of the powder after sintering. It is worth mentioning that due to the high energy of grain boundaries, the nano-crystalline or ultrafine-grained materials are generally susceptible to grain coarsening at elevated temperatures [12]. Accordingly, the high sintering temperature and long sintering time allow the powder to grow. As shown in Figure 2, the third peak is found at the diffraction angle of about 65 deg in the ODS steels containing varying amount of Y$_2$O$_3$. According to the results of XRD analysis, all diffraction peaks correspond to (Fe,Cr) phase which is the same as powder after milling. No diffraction peak of Y$_2$O$_3$ was also detected in the ODS steel with different Y$_2$O$_3$ content.

In order to investigate the effect of Y$_2$O$_3$ concentration on the microstructure of ODS steels, the SEM observations were carried out. Figure 3 shows the typical surface morphology of ODS steels after sintering at 1000 °C for 2 h.

As can be seen in Figure 3, observation under low magnification can observe the grain boundaries between particles which suggest that ODS steels with different Y$_2$O$_3$ content seem to be not fully sintered at 1000 °C for 2 h. Metallographic characterization also observes the presence of pores. Mostly, it was found in the Y$_2$O$_3$-free steel. With the increase of Y$_2$O$_3$ concentration, the porosity is likely to decrease. Moreover, it is important to note that further detail characterization at high magnification observes mainly three different areas in the sintered ODS steels: black precipitates (No. 1), bright area as matrix (No. 2) and grey areas (No. 3) as shown in Figure 3. The grey and black areas with different in size were randomly distributed in the steel matrix.

### 3.3. Structure of ODS steel after sintering at 1100 °C

Figure 4 shows the XRD profiles of ODS steels with different Y$_2$O$_3$ content after sintering at 1100 °C for 2 h.

![Figure 4. X-ray diffraction patterns of ODS steels containing 0, 0.25, 0.35 and 1wt. % Y$_2$O$_3$ after sintering at 1100 °C for 2 h.](image)

Similarly to the results as presented in Figure 2, the sample sintering at 1100 °C for 2 h leads to narrowing and increasing the XRD diffraction peaks. This is attributed to the densification of
crystallites and grain growth. The X-ray diffraction analysis also confirm that the phase of ODS steels with different Y$_2$O$_3$ content after sintering at 1100 °C is similar to that of at 1000 °C.

Figure 5 shows the surface morphology of ODS steels with different Y$_2$O$_3$ content after sintering at 1100 °C for 2 h. It can be seen that the sample sintered at 1100 °C is denser than that of at 1000 °C. The microstructural characterization also reveals that the samples consist of bright area (2) associated with small grey areas (3) and black precipitates (1), similarly to the microstructure of the sample sintered at 1000 °C for 2 h. However, the size of grey areas and black precipitates in the ODS steel sintered at 1100 °C is bigger than the previous one due to the grain growth with the increase of sintering temperature. The EDX analysis at high magnification images needs to be done to determine the composition of aforesaid structure.

![Figure 5. BSE Comp images of ODS steels containing (a) 0, (b) 0.25, (c) 0.35 and (d) 1wt.% Y$_2$O$_3$ after sintering at 1100 °C for 2 h.](image)

Moreover, unlike the ODS steel containing 0, 0.25, and 0.35 wt% Y$_2$O$_3$, the significant change in the ODS morphology is found in the ODS steel containing 1 wt% Y$_2$O$_3$. We carried out the EDX elemental mapping to observed the distribution of elements in the ODS steel containing 1 wt% Y$_2$O$_3$ with the results as presented in Figure 6.

According to the results of SEM-EDX analysis, we can clearly observe the distribution of Fe, Cr, Mn, Si and O in the dark area of BSE image. It can be conclude that some elements was somehow oxidized after sintering at 1100 °C for 2 h. This is probably due to that the dissolution of Y and O takes place [4] which leads further formation of a compound consisting of Fe, Cr, Mn, Si and O. This will significantly affects the properties of ODS steels.

### 3.4. Density and hardness

Figure 7 presents the density and hardness of ODS steels with various concentration of Y$_2$O$_3$ after sintering at 1000 °C and 1100 °C for 2 h.
Figure 6. BSE comp image and corresponding EDX elemental maps of ODS steel containing 1 wt. % Y$_2$O$_3$ after sintering at 1100 °C for 2 h.

Figure 7. (a) Density and (b) hardness of ODS steel containing varying concentration of Y$_2$O$_3$ sintered at 1000 °C and 1100 °C for 2 h.

The curve shows that the Y$_2$O$_3$-free steel exhibits low density and hardness compared to ODS steel containing Y$_2$O$_3$. The density of the samples after sintering at both temperatures has a tendency to increase with increasing of Y$_2$O$_3$ concentration, as shown in Figure 7a. In addition, a higher sintering temperature produces a denser ODS steels. However, a deviation is found in the 1 wt. % Y$_2$O$_3$ steel after sintering at 1100 °C. The density calculation clearly indicates that the sample tends to porous. The density value of present study is smaller compared to the density of ODS steels prepared by hot-isostatic press (HIP) [5, 13] and spark plasma sintering (SPS) [5]. This is mainly due to the compressive stress used in this study is lower compared to aforementioned studies. In general, a higher compressive stress results in a higher density.

On the other hand, the graph as shown in Figure 7b, reveals a steady increase in hardness of the samples (up to 0.35 wt.% Y$_2$O$_3$ concentration) in both sintering temperatures. More than 0.35 wt.% Y$_2$O$_3$, there has been a slight increase in ODS hardness after sintering at 1000 °C and a decrease in hardness after sintering at 1100 °C. Since the processing parameter for all samples is same, the
different just in the ODS steel composition, it can be assumed that the hardness increase is attributed to the denser microstructure with the increase of Y$_2$O$_3$ concentration and dispersion oxide particle in the matrix. The presence of Y$_2$O$_3$ in the steel can suppress the growth of the steel grains [2], resulting a high sample density and high sample hardness. Based on the results as presented above, the decrease of density and hardness of 1 wt.% Y$_2$O$_3$ steel after sintering at 1100 °C could be linked to the results of microstructure analysis as shown in Figure 6. It is reasonable to assume that that the microstructural change after sintering at 1100 °C for 2 h leads to decrease in density and hardness of ODS steels.

4. Conclusions
The ODS steels containing varying concentration of Y$_2$O$_3$ (0, 0.25, 0.35 and 1 wt. %) are successfully synthesized using a powder metallurgy technique at 1000 °C and 1100 °C. After sintering at the aforesaid temperatures, the ODS steels with different Y$_2$O$_3$ content are mainly composed by (Fe,Cr) phase. Metallographic characterization at high magnification indicates the distribution of precipitates in the ODS steels. Some elements are somehow preferentially oxidized in the ODS steel containing 1 wt. % Y$_2$O$_3$ after sintering at 1100 °C to form a compound consisting of Fe, Cr, Mn, Si and O. The ODS steels sintered at 1100 °C exhibited a higher density and hardness compared to the samples sintered at 1000 °C. In addition, the density and hardness of the sample increase with increasing of Y$_2$O$_3$ concentration. However, further increase in Y$_2$O$_3$ concentration (1 wt. %) leads to decrease in density and hardness of ODS steels.

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