Microscopy of Alloy Formation on Arc Plasma Sintered Oxide Dispersion Strengthen (ODS) Steel

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Abstract. The oxide dispersed strengthened (ODS) alloys steel developed as structure material for nuclear power plants (NPP) has good resistant against creep due to their unique microstructure. Microscopy investigation on the microstructure formation during alloying process especially at the early stages was carried out to study the correlation between structure and property of ODS alloys. This was possible thanks to the arc plasma sintering (APS) device which can simulate the time dependent alloying processes. The ODS sample with composition of 88 wt.% Fe and 12 wt.% Cr powder dispersed with 1 wt.% ZrO2 nano powder was mixed in a high energy milling, isostatic compressed to form sample coins and then alloyed in APS. The Scanning Electron Microscope (SEM) with X-ray Diffraction Spectroscopy (EDX) line scan and mapping was used to characterize the microstructure and elemental composition distribution of the samples. The alloying process with unification of each Fe and Cr phase continued by the alloying formation of Fe-Cr by inter-diffusion of both Fe and Cr and followed by the improvement of the mechanical properties of hardness.

1. Introduction.

Research on special high temperature materials plays an important role in supporting the national nuclear energy program. Meanwhile due to its superior properties at high temperature, high chromium oxide dispersed strengthened (ODS) alloys are believed to be the best candidate as structure material for nuclear power plant (NPP) of 4th generation [1,2]. Their origin can be found especially on their good resistant against oxidation and creep at elevated temperature. This is because of their unique microstructure that formed during alloying process. However, in this case the early stages of phase formation must play a critical role. Therefore, the mechanism of alloying process of the ODS steel was necessary investigated to get reliably and accurate information that is important for improving the performance of the alloy manufacturing. Commercial ODS steels are usually added by the Yttrium oxide Y2O3 particles up to 1 wt% [3]. The zirconia particles are, however, more proposed for application as nuclear structure material because of their low neutron absorption cross section. On the other hand, the addition of zirconium to a ferritic matrix of ODS steel could also effectively improve the performance against high temperature and corrosion by the formation of more stable Y-Zr oxide [4,5]. Manufacturing of ODS steels was normally performed via powder metallurgical route which can easily disperse the oxide particles homogeneously. However, this process is limited by the amount of material can be added, costly and very time consuming [6]. Therefore, many researches was worldwide conducted in developing of new manufacturing technique which all are aimed in finding of processes that are faster and lower in cost. A casting route will be the most effective in accordance to the cost and production capacity, but it exhibits difficulties in uniform dispersion of the ceramic particles in liquid metal for the chemical reactions with the matrix elements [7]. Recent method using Spark Plasma Sintering (SPS) can obviously produce ODS alloy with quite good quality by relatively fast process time, but this technology still need high process energy [8]. In this work the proposed new method of Arc Plasma Sintering (APS) which uses plasma as heat source has been developed in PSTBM-BATAN can significantly reduce process time and energy consumption. This apparatus is used for synthesizing of alloys with different composition. Recent work reported the fabrication of Fe-Cr and feritic ODS alloys to obtain the optimal properties on the high temperature material application [9]. In this work, microscopy investigation on the alloying process especially at the first time of their formation during ODS steel production was performed with help of arc plasma sintering (APS) with the aim to study the possible correlation between structure and property of ODS alloys.

2. Experimental

For the experiment the samples composed of 88 wt.% Fe and 12 wt.% Cr powder with addition of 0.5 and 1 wt.% ZrO2 nano powder as dispersoids were prepared. The powder were mixed in a rotary mill for 3 hours and were isostatic pressed with 20 tons of load producing sample coins of 15 mm of diameter and 4 mm thick. The sample coins were then consolidated in APS at 12 V and 25 Amps with time variation ranges between 2 and 4 minutes. The technical drawing of the experimental set up in APS is shown schematically in Fig. 1. In this work the F-Cr ODS ferritic steel was developed with 12 wt.% Cr and 0.5 and 1 wt.% of the dispersion particles ZrO_2. The samples were prepared by mixing and milling all the materials (Fe, Cr and ZrO_2 powder) for 1, 2 and 3 hours. After milling the samples were pressed by isostatic compress machine with the compression load of 20 Ton to form sample coins. The samples were then consolidated by sintering in the APS for 4 minutes. The principal design of the APS is illustrated schematically in Fig. 1 [6]. The process was carried out in fully argon atmosphere in order to protect against oxidation. The sample coin was placed on a copper cup and exposed by the plasma with the power 300 Watt and variation of time.

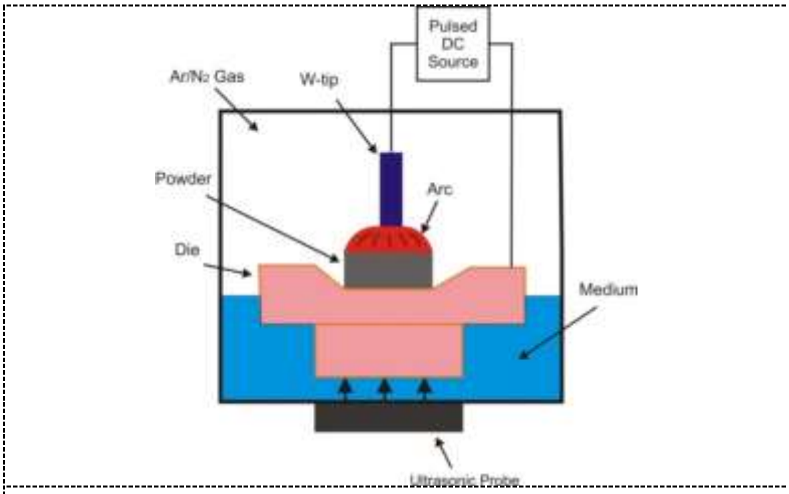

Figure 1. Schematic diagram of APS

APS is an experimental sintering and melting device newly developed at the Centre of Science and Technology for Advanced Materials (PSTBM) of Indonesia nuclear energy agency [10]. Its heart is an arc plasma source, which can provide a very high process temperature in a short time. This allows a fast process of densification or consolidation of almost every powder metallurgical prepared samples. Therefore, APS is suitable for simulation of some thermal induced processes such as alloy formation. The Scanning Electron Microscope (SEM) equipped with X-ray Diffraction Spectroscopy (EDX) was used to characterize the microstructure and elemental composition of the samples. EDX line scan and area mapping were applied in order to visualize the elemental distribution in the sample. The mechanical properties of the samples were measured by means of Vickers micro hardness.

3. Results and Discussion

Figure 2 shows the SEM image of the sample after compression 20 tons. One can see clearly every single Fe and Cr particle. The result of the press process is quite good. The amount of porosity is about less than 15%.

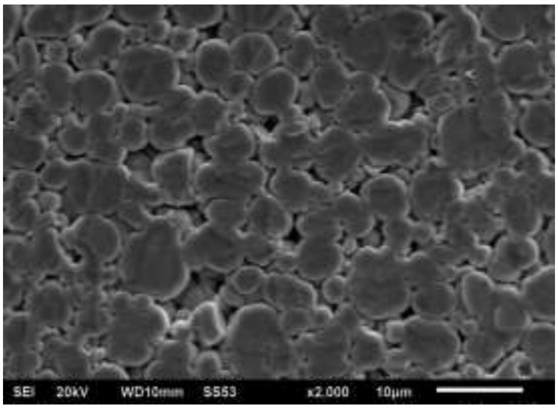

Figure 2. SEM image of the sample after compression 20 tons

The microstructure of ODS ferritic alloys dispersed by 0.5 and 1 wt.% were observed by SEM. The results are illustrated in Figure 2. The SEM micrograph in Figure 3a shows general appearance of the sample microstructure after 4 minutes consolidation through sintering in APS taken by SEM at 20 kV using backscattered electrons detector (BSE). The sample consists of two phases, the white matrix phase and large precipitate phase. The porosities have been completely disappears. From the EDX analysis it is obvious that the matrix (zone 1) mainly consists of iron phase with 96.68 wt.% Fe and 1.60% Cr. While, the zone 2 is chromium phase dominated by 96.51 % Cr and Fe 1.85 %. This is assumed to be caused by the similarity of Gibbs free energy of formation of single elements compared to those of two and more elements. The EDX line scan in Figure 3b was put along the straight line as indicated in Figure 3a showing the compositional changes of the Fe and Cr in the phase interface. It can be observed that the distribution graph of both Fe and Cr exhibits characteristic diffusion zone, which elucidate the diffusion in two directions. Fe and Cr are moving into each other. However, the concave flow of the graph explains that the Cr diffusion must be faster than Fe. From the result can be concluded that the formation of Fe-Cr alloy begins by the melting of each alloy element forming the Fe and Cr phases then continued with the formation of Fe-Cr alloy which is determined by the diffusion process. This diffusion process is possible because of the existence of the relatively high chemical potential due long range elemental in homogeneity in the Fe matrix and Cr phase.

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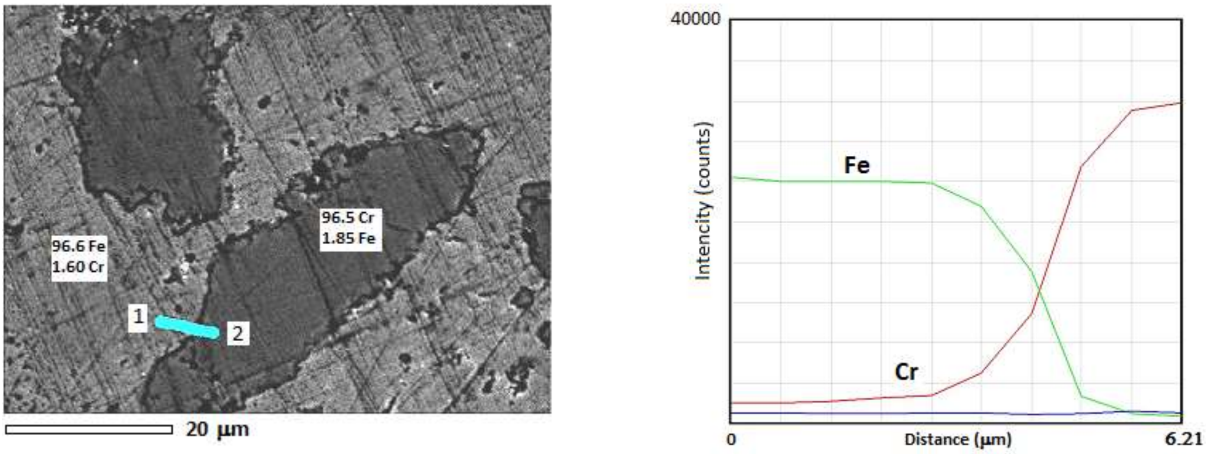

a. **b.**

Figure 3. SEM image of ODS steel produced by APS (a) line scan mapping (b)

The behavior of the alloy sintered by APS differ significantly to the results of conventional sintering using the sintering furnace as shown in Figure 4.

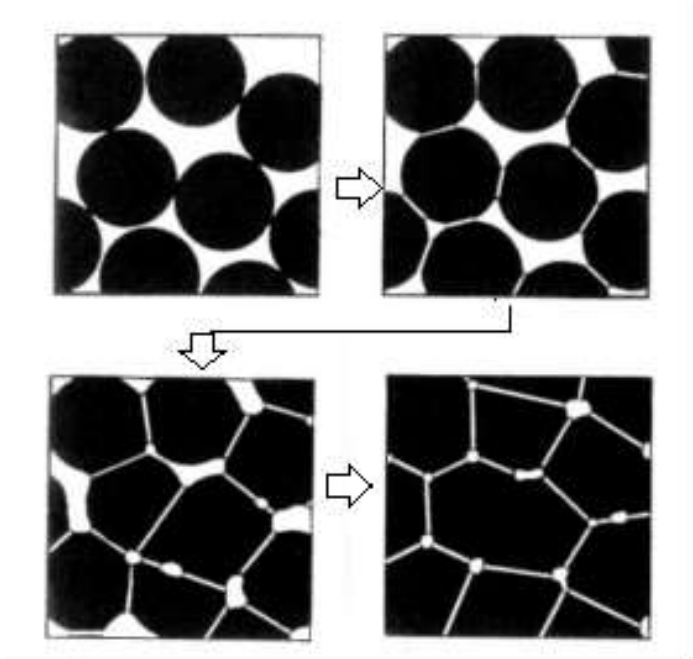

Figure 4. Alloying scheme of conventional sintering process

The early portion of sintering produce bonds between contacting particle and focuses on the growth of interparticle bonds, followed by densification and pore elimination. The process was continued by the diffusion processes at sintering temperature that cause necks to form and grow the grain. The zirconia was finely dispersed in the matrix and in the grain boundaries of both Fe and Cr phase as well. From the observation on all the samples it is revealed that the alloying process was strongly depend on the sintering time. However, after 4 minutes sintering showed the more proper alloy of Fe with Cr dispersed phase. The hardness of 150 VHN after 4 minutes of sintering was achieved showing the improvement of the mechanical properties caused by the alloying process during sintering process.

4. Conclusion

An experimental study on the alloy formation during early stages of their formation using APS has been performed. The alloying process begins with unification of each Fe and Cr phase due to preferred low Gibbs free energy for the formation of phase of the same atoms. With increasing time, the development of Fe-Cr alloy formation in dependency on sintering time. Alloy formation is the result of inter-diffusion of both Fe and Cr in both directions forming zone of Fe-Cr phase, however Cr moves faster into the new formed phase than Fe. These development followed by the improvement of the mechanical properties that can be observed on the hardness of 150 VHN after 4 minutes of sintering.

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