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# High temperature Oxidation of ODS alloy with zirconia dispersions synthesized using Arc Plasma Sintering

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**Abstract.** Microstructure formation and oxidation behaviour of the Oxide Dispersion Strengthened (ODS) steels for application as structure material in Nuclear Power Plant was investigated. A mixture composed of Fe and 12 wt. % Cr powder with addition of 0.5 and 1 wt.% ZrO<sub>2</sub> particles was milled and isostatic pressed to form a sample coin. The coin was then consolidated in the Arc Plasma Sintering (APS) for 4 minutes. The samples were subjected to the high temperature oxidation test in the Magnetic Suspension Balance (MSB). The oxidation test was carried out at 700°C for 6 hours to evaluate the oxide growth in the early stage of its formation by extraction the mass gain curve. The Scanning Electron Microscope (SEM) imaging and X-ray Diffraction Spectroscopy (EDX) elemental mapping were performed to study the microstructure change and compositional distribution. SEM and EDX observation revealed the time dependent development of the Fe-Cr-phases during consolidation. The oxidation rate behaviour of the samples followed the parabolic rate characteristic for inward oxidation process driven by oxygen inward diffusion through the oxide scale with the maximum weight gain around of 60 g/m<sup>2</sup>. The oxidation resistance was strongly affected by the formation of the oxide protective layer on the surface. In so far, addition of zirconia particles has played no significant role to the oxidation behaviour.

## 1. Introduction

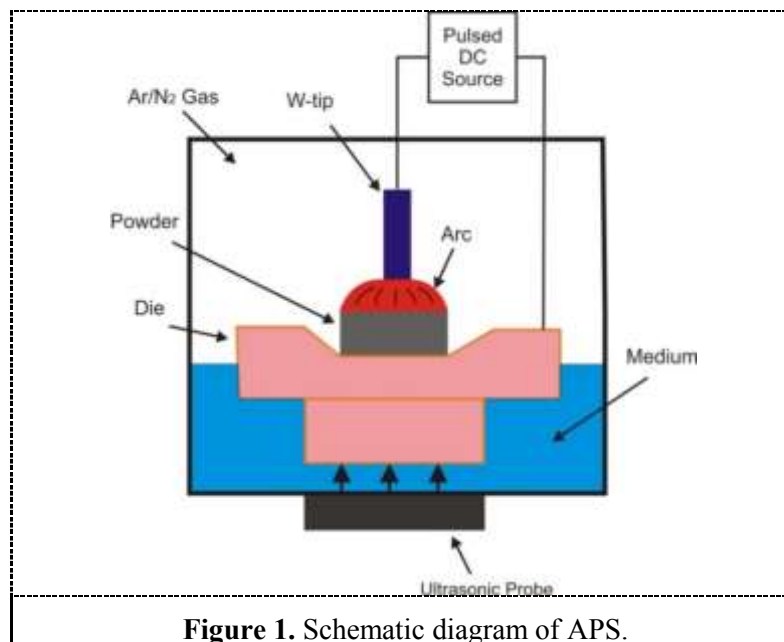
Oxide dispersion strengthening (ODS) of high chrome (Cr) steel is a promising way to get materials for high temperature application. Recently, ODS ferritic steels were developed as material of the 4th generation of high temperature nuclear power reactor due to their excellent strength and creep properties [1]. However, according to the environment of high temperature reactor application, the material should also be designed to address the problem of oxidation and irradiation damage. The dispersion of zirconia was used to replace the Ytria for synthesizing of ODS ferritic steel applied as nuclear reactor components because of the low cross section for neutron absorption of zirconium and the good corrosion resistance [2]. Previous work has concluded that addition of zirconia into a ferritic Fe-14Cr-5Al-3W-0.4Ti-0.25Y<sub>2</sub>O<sub>3</sub> matrix could effectively lead in better performance of the ODS steels against high temperature and corrosion [3, 4]. Recently, some method for ODS steel manufacturing was also developed to improve the powder metallurgical route process which is limited by the amount of material and relatively high cost. The new method we used in this work for sintering of high temperature materials, Arc Plasma Sintering (APS), developed in PSTBM-BATAN has been successfully reduced time and energy consumption. This device was used for synthesizing many high temperature alloys such as Fe-Cr and the ferritic ODS steels with addition of nano Ytria and zirconia particles dispersed along the alloy matrix to obtain the optimal properties on the high temperature material application [5]. In this work, oxide dispersion strengthened (ODS) ferritic steel with nominal



composition of Chromium as high as 12 wt.% dispersed with 0.5 and 1 wt.% ZrO<sub>2</sub> was synthesized by the Arc Plasma Sintering (APS) method. The microstructure and the oxidation resistance were investigated to determine the oxidation characteristic concerning to the zirconia addition on the ODS ferritic steel.

## 2. Experimental

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<sub>2</sub>. The samples were prepared by mixing and milling all the materials (Fe, Cr and ZrO<sub>2</sub> 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.

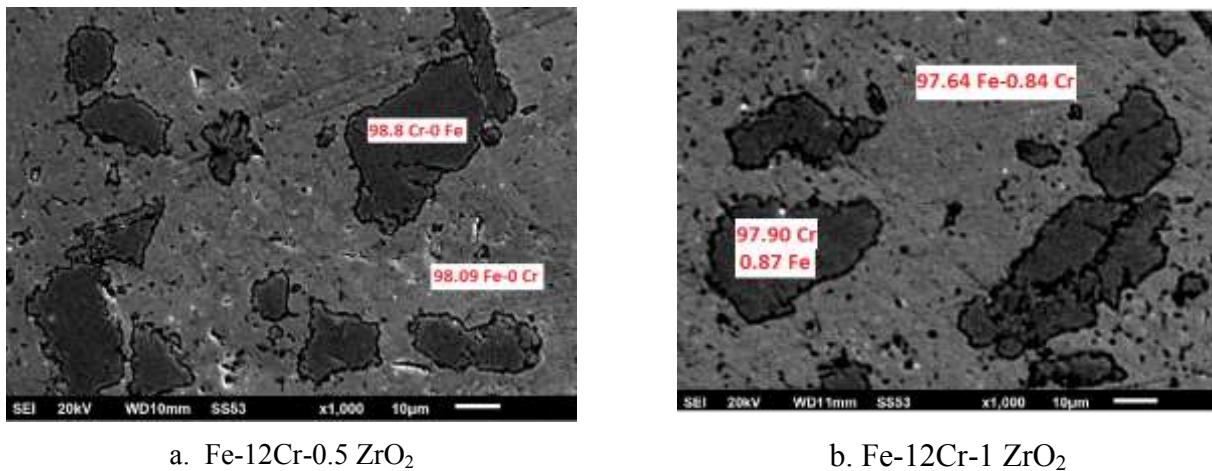


The morphology and composition of the alloys were studied by means of Scanning Electron Microscopy (SEM) coupled to Energy Dispersive Spectroscopy (EDX). The EDX spectra illustrate the chemical composition of the samples. The mechanical properties of the consolidated samples were studied using the Vickers micro hardness (HV) measurement. The high temperature oxidation test was performed in the Magnetic Suspension Balance (MSB) workstation in air atmosphere at 700 °C to simulate the temperature in normal reactor operation. Detail information about MSB test is described elsewhere [7]. The oxidation time was carried out for 6 hours to get insight in to the early stage of oxide formation. The oxidation characteristic was extracted from the curve describing the relation of the weight gain to the oxidation time. The oxide scale and the effect to the mechanical properties were evaluated based on the microstructure and hardness measurements as described above.

## 3. Results and Discussion

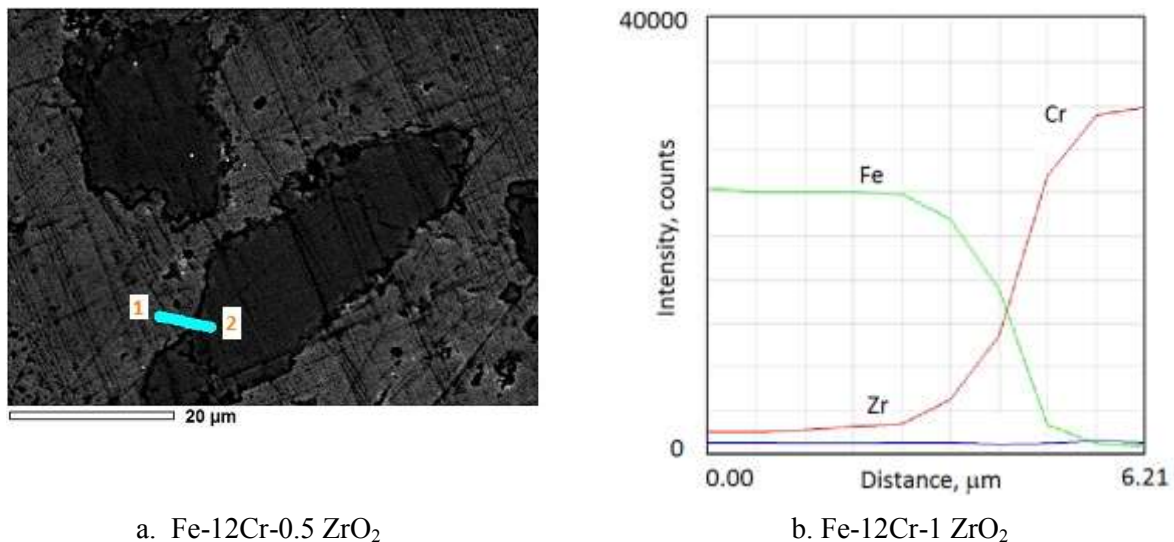
### 3.1. Microstructure

The microstructure of ODS ferritic alloys dispersed by 0.5 and 1 wt.% were observed by SEM. The results are illustrated in Figure 2.



**Figure 2.** SEM images of the ODS steel milled 1 hour and sintered for 4 minutes.

The SEM images in Fig. 2 show the alloy microstructure consisting of bright matrix which dominated by Fe up to 98 wt.% and the dark second phases which is Cr-rich, about 98.8 wt.%. This observation can be concluded that the alloy formation begins by the formation of two single primary Fe and Cr phases. However, there are no significant differences between the sample with and without zirconia addition. Further, elemental transition from Fe to Cr phase interface is described by the results of EDX line scanning for the alloy with 0.5 % zirconia dispersion with milling time of 3 hours, shown in Figure 3 [8].



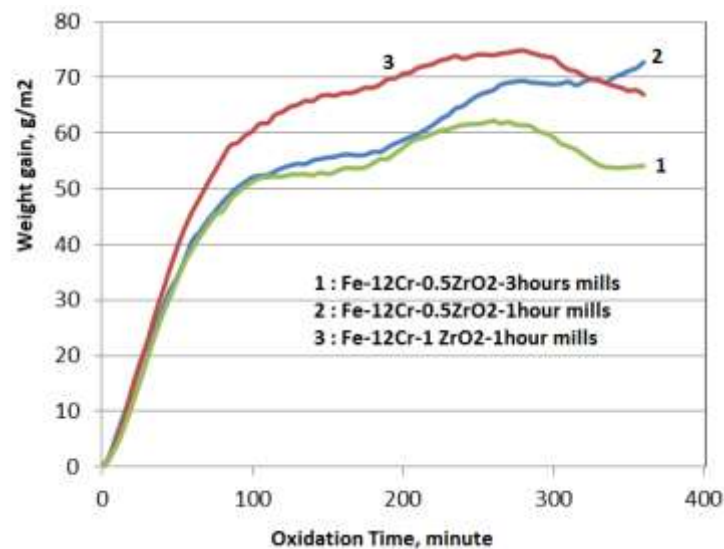
**Figure 3.** EDX line scan in the Fe-Cr phase interface

Based on the observation in Fig. 3, as expected the alloy formation is the result of inter-diffusion of both elements Fe and Cr in both direction forming a diffusion zone. However Cr moves faster into the new formed phase than Fe, as can be seen from the graphic showing concave structure. Both side of the phase exists. Detailed of microstructure and the alloying process was explained by the diffusion theory of Fe and Cr [6, 8]. The zirconia was finely dispersed in the matrix and in the grain boundary of

both Fe and Cr phase as well. These development followed by the improvement of the mechanical properties that can be observed on the maximal hardness of 150 VHN after 4 minutes of sintering for sample of Fe-12Cr-0.5ZrO<sub>2</sub> with milling time for 3 hours.

### 3.2. Oxidation behavior

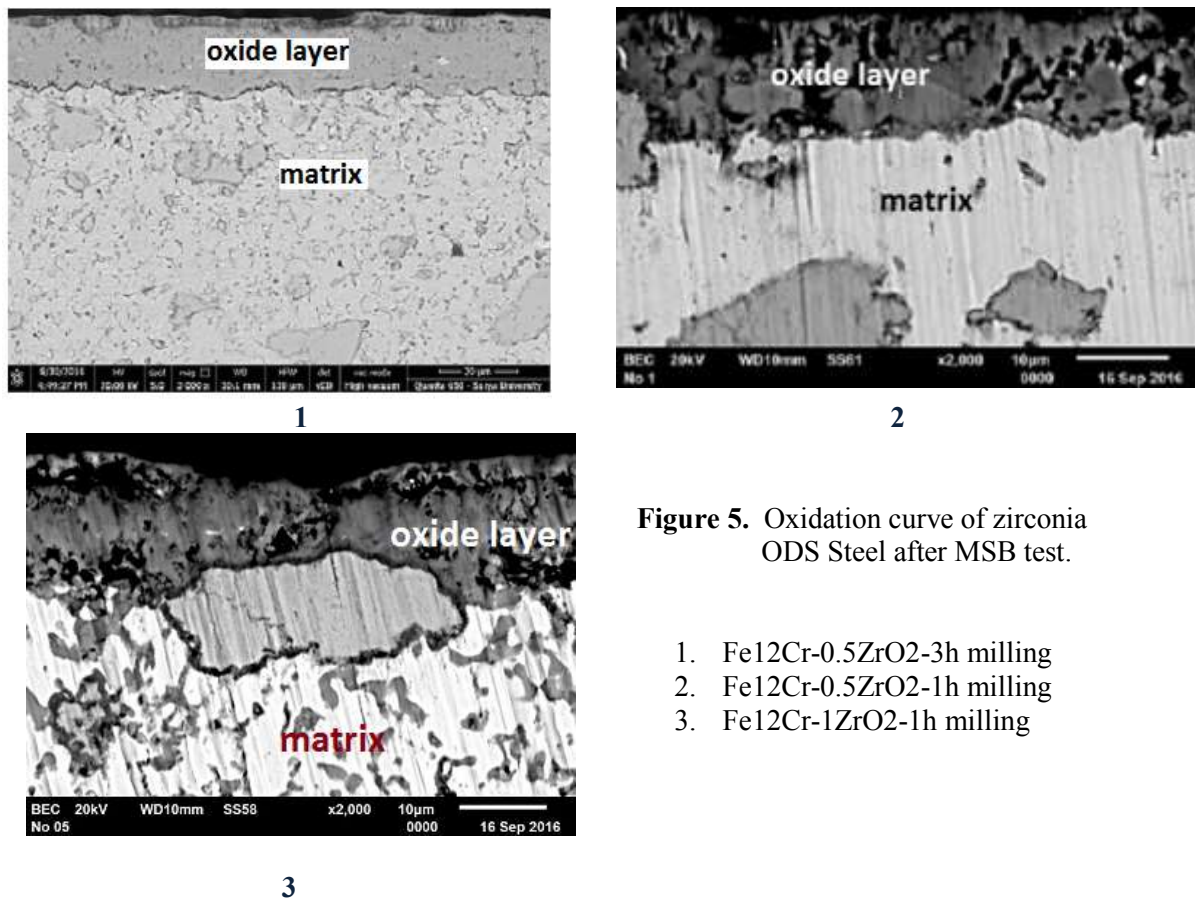
Figure 4 shows the oxidation characteristic curves of ODS steels with zirconia dispersion oxidized in air at 700°C for 6 hours.



**Figure 4.** Oxidation curve of zirconia ODS Steel after MSB test.

The weight gains resulted from the oxidation in air are plotted as a function of the oxidation time. The early stage of oxidation during heating up to 150 minutes showed the oxidation rate that follows parabolic growth kinetics where the mass gain is proportional to the square root of time. This early stage was the most important stage that determine the total weight gain which elucidate the formation behaviour of the protective oxide layer. The characteristic of sample that follows the parabolic kinetic explains that the oxygen inward diffusion is the driven process during the formation of the oxide scale on the top of the sample surface. This parabolic characteristic stands in agreement with the well-known high temperature oxidation characteristic of Fe-Cr alloy [9]. The mechanism of high-temperature oxidation of Fe-Cr alloy is widely accepted to be the diffusion of oxygen anions through the iron oxide lattice [10]. The metal cations transport however is fully inhibited. The milling time seems to be not significantly influence the oxidation behavior, as obvious from the the oxidation curve. However, addition of zirconia particles 0.5 wt.% is likely to affect the weight gain. The weight gain of the samples with 0.5 wt.% ZrO<sub>2</sub> show significant decrease compared to sample containing 1 wt.% ZrO<sub>2</sub>. This leads to the fact that the first two samples show higher oxidation resistance.

The result of the SEM analysis in Fig 5 shows the sample cross-sections revealing the oxide scales formed during oxidation test.



**Figure 5.** Oxidation curve of zirconia ODS Steel after MSB test.

1. Fe12Cr-0.5ZrO<sub>2</sub>-3h milling
2. Fe12Cr-0.5ZrO<sub>2</sub>-1h milling
3. Fe12Cr-1ZrO<sub>2</sub>-1h milling

From the EDX result the oxide layer is identified as stable Fe<sub>2</sub>O<sub>3</sub>. The thickness of the oxide layers of the samples around 15-20 μm is believed to be a protective layer and explains the oxidation behaviour as shown in Fig. 3. Based on the layer thickness and oxidation behaviour of some Fe-Cr oxidation [9, 10], showed that the layer thickness and oxidation rate of the APS sintering product was good enough for high temperature corrosion behavior.

#### 4. Conclusion

From this study, it can be concluded that APS experiment can describe the alloy formation sufficiently. The alloying of the ODS steel was inter diffusion process of Fe and Cr element that followed by the increasing of hardness. During the first stage of heating at 700°C, the oxidation behaviour of the ODS followed the parabolic growth kinetic. Addition of low concentration of zirconia in the alloy decreases the oxidation, whereas the addition of zirconia of higher concentration does not influence the oxidation rate. The effect may be caused by the influence of diffusion process due to the presence of nano oxide particle which is modify the oxide scale in the manner that decreases the oxygen diffusion channel. The oxide layer formed on the surface is a stable Cr<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>.

#### Acknowledgment

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