

Effect Of cobalt addition on Fe-20Cr-5Al-1Y₂O₃ ODS alloy fabricated by arc plasma sintering (APS) process

B Suharno¹, D E Jodi¹, N R Pratama¹, A Dimyati²

¹ Department of Metallurgy and Materials Engineering, Faculty of Engineering
University of Indonesia, Depok 16424, Indonesia

² Center for Science and Technology of Advanced Materials. BATAN
Puspiptek Area, Serpong, Tangerang 15314, Indonesia

prof.suharno@gmail.com

Abstract. In this research, microstructure, phases and hardness of Fe-Cr-Al based Oxide Dispersion Strengthened (ODS) alloy with addition of cobalt (Co) produced using arc plasma sintering (APS) method were investigated. ODS alloy has an important role particularly in high temperature applications, since it has the ability to maintain their strength at high service temperature. On the other hand Co is well known to increase creep resistance of many alloys, which could in turn increases their lifetime in high temperature environment. To produce ODS alloy, usually high cost and long processing time are required. To overcome these problems, a new sintering method based on arc plasma was used in this study, which has been successfully applied in previous works. The alloys composed of Fe-20Cr-5Al-1Y₂O₃ with addition of 0.5, 1, 1.5 and 2 wt% cobalt were first prepared by mechanical alloying (MA) comprising high-energy milling for 8 hours and subsequent isostatic pressing at 20 ton-force/in² prior to sintering by APS method up to 28 minutes. The sintered samples then characterized by means of Scanning Electron Microscopy (SEM) equipped with an Energy Dispersive X-Ray Spectrometer (EDX) to observe the microstructure, the X-Ray Diffraction Technique (XRD) to investigate the formed phases, and Vickers microhardness test to examine hardness value of the alloys. The results show that APS method is technically and economically feasible for fabricating ODS alloys since this process significantly reduces time and energy required. Variation of cobalt in the alloy obviously does not cause observable microstructure difference. However, the alloy with 0.5 wt% cobalt exhibited single FeCrAl matrix phase, while at higher cobalt concentration additional AlCo phases were found in the FeCrAl matrix, which can be assumed to improve the high temperature usage of the alloy. Cobalt addition also increased hardness value of these alloys.

1. Introduction

Oxide dispersion strengthened (ODS) alloys are promising material mainly for high temperature and corrosive applications, i.e. nuclear reactor and power generation plant [1, 2], because of the presence of oxide particles that are dispersed on grain and grain boundaries that could inhibit creep during high temperature applications [3]. The presence of chromium and aluminum in Fe-20Cr-5Al alloy help to improve its oxidation resistance due to the formation of alumina scales, whereas the addition of 1 wt% Y₂O₃ improves both the mechanical strength and the high temperature properties, and also lowers the minimum aluminum concentration required for the formation of protective alumina scale on alloy



surface [4, 5]. The addition of cobalt in alloys, generally improves tensile strength, but it also helps improving fatigue and creep resistance [6].

ODS alloys are commonly manufactured using powder metallurgical technique and subsequent sintering in a conventional heating furnace, which is constrained by the high cost and it is also very time consuming. To overcome these issues, a new method for sintering ODS alloy and other high temperature alloys based on plasma generated by an electrical arc has been developed in Center for Science and Technology of Advance Materials (PSTBM) BATAN which is known as Arc Plasma Sintering. APS method for producing Fe-Cr with variation of Cr content and with addition of ZrO_2 has been proved to be an effective sintering method with less time and energy required [7]. This method also has been previously studied for the fabrication of silicon carbide, which resulted in an increase of mechanical strength followed by lower energy and less time required for its sintering process [8].

This study's primary objective is to investigate the effect of cobalt content variation in the range of between 0.5 and 2 wt% on the microstructure, phase formation and mechanical property of the Fe-20Cr-5Al-1Y₂O₃ ODS alloy after sintering by the APS method.

2. Experimentals

Four samples of ODS Fe-20Cr-5Al-1Y₂O₃ alloy were fabricated from Fe with a purity of 99.9%, Cr of 99%- and Al of 99% purity, all starting materials are powder, with cobalt addition which was varied 0.5, 1, 1.5, and 2 wt% firstly by means of powder metallurgy. The initial powder size was measured and presented in Table 1. The powder mix was mechanically alloyed using high-energy mill HEM E3-D for 8 hours with ball to powder ratio of 2:1 in order to reduce powder size and to homogenize the powder mixing. The powder was then pressed using uniaxial pressing method with pressure of 20 ton-force/in² to produce sample coin as green material. Sintering process is performed using APS method up to 28 minutes at 12 Volt and 80 Amperes to consolidate the samples. The morphology and composition of the alloys were observed by JEOL SEM-EDX. Investigation of formed phase was further investigated by RIGAKU-SMARTLAB X-Ray Diffraction (XRD), and mechanical properties were measured using Vickers microhardness (HV) tester using 300 gf load and 10s indentation time.

Table 1. Initial powder size of Fe, Cr, Al, Y₂O₃, and Co.

Powder	Fe	Cr	Al	Y ₂ O ₃	Co
Average Powder Size	5 μ m	5 μ m	20 μ m	50 nm	5 μ m

3. Results and discussion

3.1. Microstructures

The microstructures of the specimens are observed using optical microscope (OM) and SEM, which are shown in Figure 1 and 2. Based on the microstructure observation, the addition of cobalt in the ODS alloy alloys seems to give no significant difference. The sharp-angle grains in the microstructure in all samples were mainly caused by the impact force that occurred during high energy mechanical alloying process, in which the powder was work hardened and fractured as the result of fatigue mechanism, and the powder turn to flaky shape with sharp angles [9, 10].

There is also no significant microstructure and grain size changes with the cobalt content. The average grain size for each ODS alloy sample with different cobalt content is shown in Table 2, the grain size has obviously been increased significantly compared with initial powder used in this study.

In addition, the increasing cobalt content also does not yield any significant changes in the number and size of porosities. All of the specimens exhibit localized and reduced diameter in porosity size and shape, which is obviously caused by the compaction and the sintering process [11, 12].

To observe the relationship between cobalt content and its dispersion in the alloys, SEM and EDX measurement were used. EDX examination was performed on the sample with 0.5 and 2 wt% cobalt addition, the results are shown in Figure 3 and Table 3.

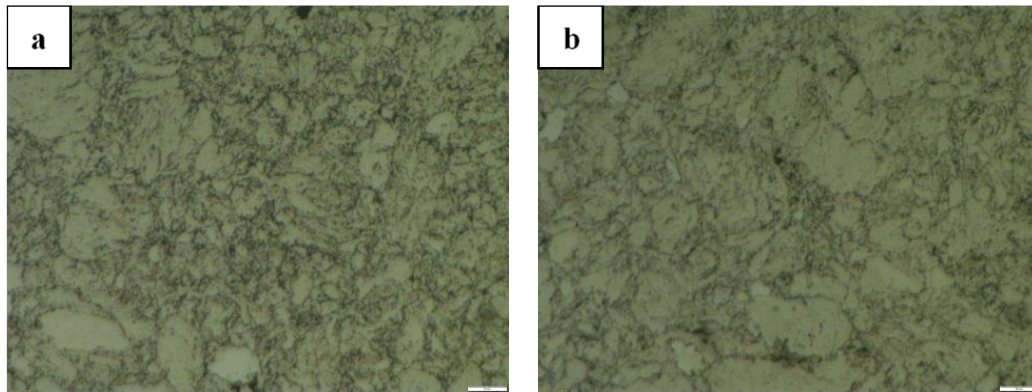


Figure 1. OM micrographs of the arc plasma sintered Fe-20Cr-5Al-1Y₂O₃ ODS alloy observed at 1000x magnification, with addition of (a) 1 and (b) 1.5 wt% Co.

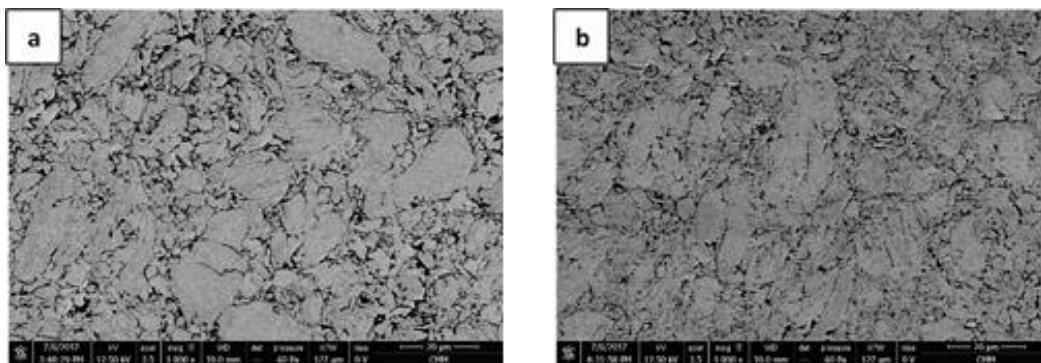


Figure 2. SEM micrographs of the arc plasma sintered Fe-20Cr-5Al-1Y₂O₃ ODS alloy with addition of (a) 0.5 wt% Co (b) 2 wt% Co.

Table 2. Average grain size for each ODS alloy sample after arc plasma sintering process.

Composition	0.5 wt% Co	1 wt% Co	1.5 wt% Co	2 wt% Co
Average grain size	12.86 μm	13.14 μm	13.77 μm	13.37 μm

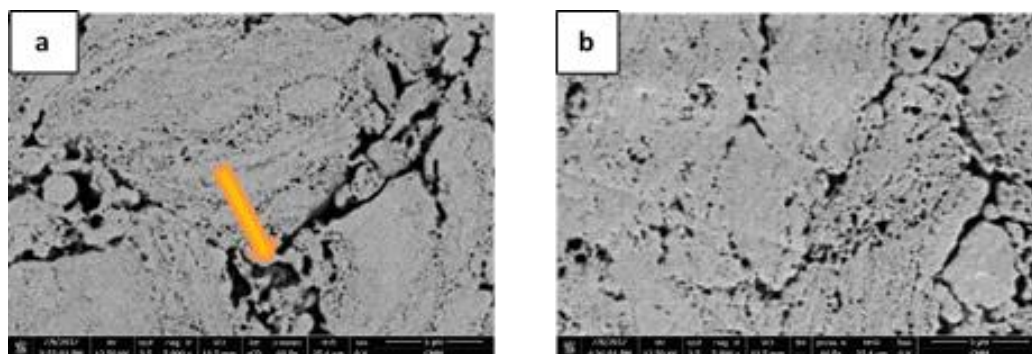


Figure 3. SEM images with the area of EDX examination on Fe- 20Cr- 5Al- 1Y₂O₃ ODS alloys samples with addition of (a) 0.5 and (b) 2 wt% Co, indicated with arrows.

From EDX results as shown in Table 3, in general, it is similar to the main composition used in the alloys, with the main elements consisting of Fe and Cr. A unique feature was found in the Fe-20Cr-5Al-

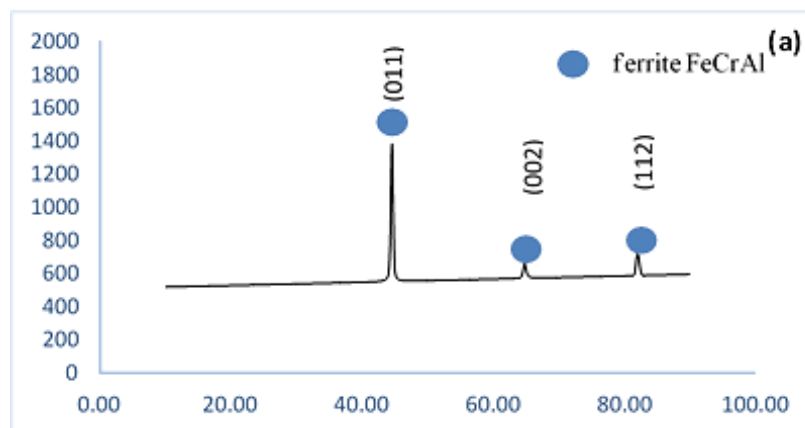
1Y₂O₃-0.5Co specimen, shown by the yellow arrow, the phase consists of Y and Al in higher composition compared to EDX result in general position which is Fe-rich, and makes the color of this phase tends to be darker. The location of this yttrium-containing phase itself is on the edge of the grain, which is caused by the original trait of Y₂O₃ that is located on the grain boundary that would help to impede the creep movement [13]. Also, from the EDX result, there is a presence of oxygen in the microstructure, which could be the oxide phase that was formed during the sample preparation before EDX, and oxygen may react with chromium and formed Cr₂O₃. This formed oxide also suppress the densification process by providing a barrier to atomic diffusion between particles, resulting in lower density value and lower mechanical strength [14, 15]. Further investigation about the formed phase will be described in the next section.

Table 3. Chemical composition of the arc plasma sintered Fe-20Cr-5Al-1Y₂O₃ ODS alloys with addition of 0.5 and 2 wt% Co measured by EDX.

Alloy	Fe (%)	Cr	Al	Co	O
0.5 Co	70.81	17.78	4.91	1.44	5.06
2 Co	70.9	20.2	2.4	4.1	2.4

3.2 Phase formation

X-ray diffraction (XRD) is used to identify phases that exist in the specimens. Results are shown in figure 4. From the XRD results, for alloy with the addition of 0.5 wt% cobalt, there are 3 main diffraction peaks at the 2 θ angular positions of 44.56°, 64.84°, and 82.01°, which are identified as ferrite phase that is rich in iron, chromium, and aluminium, since these peaks closely resemble this phase's Miller Indices and also yields the lattice parameter of 2.87 Å°. The main reason of the formation of this ferrite phase is because the main elements of this specimen is α -Fe and Cr, which cover 93.5% of total composition in the specimen. Also, the addition of 0.5% cobalt could be assumed to be too low to form any intermetallic phase of cobalt. For alloy with 2 wt% cobalt addition, besides the three main peaks that are found in alloy with 0.5 wt% cobalt, there are also three small peaks are observed at 2 θ of 31.10°, 55.35°, and 73.69° respectively, which could be identified as AlCo phase, since the angular positions closely correspond to the AlCo reflection angles, as well as its Miller Indices number, and also its lattice parameter with a value of 2.862 Å°. This intermetallic compound has the ability for high temperature applications, since this compound has high melting temperature, moderate oxidation resistance, and the ability to maintain its crystal structure up to its melting temperature [16].



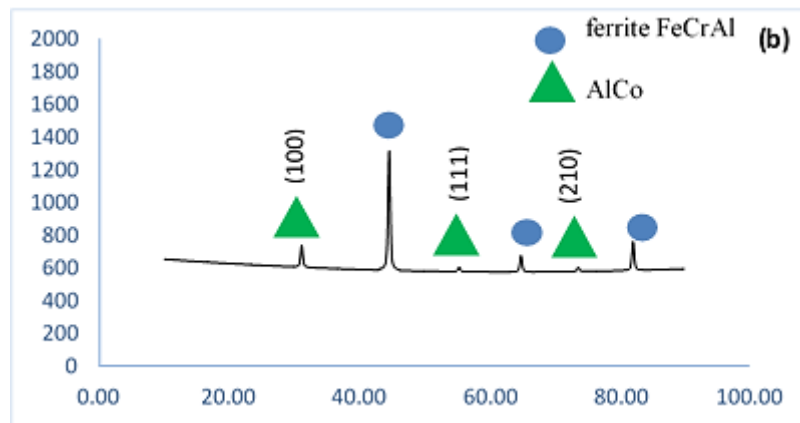


Figure 4. XRD spectren of ODS Fe-20Cr-5Al-1Y₂O₃ alloy with addition of (a) 0.5 and (b) 2 wt% Co.

Although theoretically AlCo phase is formed when the composition is around Al-65%Co to Al-73%Co, this AlCo phase could also be formed with a lower amount of cobalt when processed using mechanical alloying method, due to composition change of the powder mixture during milling process and also the enhanced solubility of AlCo phase by milling [17]. Also, in this investigation, the oxide phase that is predicted to be present on the alloys is predicted to be too low in composition, since there is no oxide phase that is detected in the microstructure. Further research is needed to understand the effect of AlCo phase on the ODS Fe-20Cr-5Al-1Y₂O₃ alloy and also the correlation between AlCo phase and Y₂O₃ particles in high temperature applications.

3.3 Mechanical properties

Figure 5 shows the Vickers microhardness results after arc plasma sintering process as a function of cobalt content. Hardness value increases with an increase in cobalt addition, which mainly affected by the addition of cobalt in ODS alloys. This result is similar to the results obtained in other previous experiments, and the reason is because Co helps to decrease the stacking fault energy and increase the plastic deformation resistance, although this behavior tends to significantly affective for face-centered cubic metals [18]. The increase of hardness in these alloys couldn't be fully associated with the presence of AlCo phase in the microstructure, but the hardness value of these alloys could reach higher number if the sintering process had been done in fully argon or vacuum atmosphere, since there's no oxide phase that would present in the specimens that could decrease the hardness value. Changes in the hardness value also affect the mechanical strength, since both of these aspects are related to each other, and increase in hardness value should also increase the mechanical strength of these alloys.

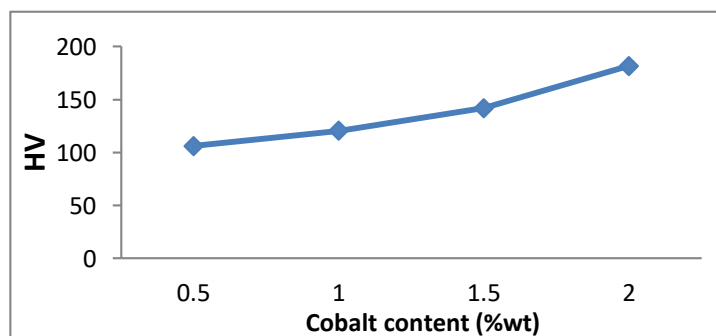


Figure 5. Vickers microhardness after arc plasma sintering vs. cobalt content.

4. Conclusion

This research has successfully fabricated alloys of ODS Fe-20Cr-5Al-1Y₂O₃ with cobalt addition using arc plasma sintering method, which is proved to reduce fabrication time, cost, and energy significantly. The addition of cobalt in 0.5 wt% in this alloy form FeCrAl main phase, and as cobalt content increase in around 2 wt%, AlCo starts to form in Fe-Cr matrix that could help this alloy in high temperature application, although further research are needed to observe the actual effect. Increase in cobalt also increase the hardness and mechanical strength value of these ODS alloys.

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