THE IMPROVEMENT OF OXIDATION BEHAVIOR OF POWDER METALLURGY MADE FE-CR ALLOYS

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Introduction

Iron chromium base alloy has been used as a plate type interconnector of solid electrolyte fuel cell (SOFC) due to its low material cost and good high temperature oxidation resistance and compatibility of thermal expansion coefficient with solid electrolyte [1-3]. The saving of manufacture process and material loss for metallic interconnector is one of solution to decrease the cost of fuel cell further.

Recently metallic interconnector has announced to be processed by powder metallurgy [4]; nevertheless few information of oxidation resistance has been given to its product.

It has been found in our study that the oxidation resistance of powder sintered plate has inferior high temperature oxidation resistance comparing to normal wrought product due to the increase of surface area of porosity.

Therefore improving the oxidation resistance of powder metallurgy made of FeCr alloy is the object of this study. The behavior of oxidation was examined at high temperature with different sintered conditions of FeCr alloys powder.

The powder was made with rotating electrode powder making technique in a range of size from 100 to 150 μm .

Experimental

The detail of self-made rotating electrode powder making equipment could refer to our published paper [5].

The cathode is made of tungsten bar with diameters of 10 mm and the anode is made of stainless steel 440(Fe-17Cr-1Mn)bar with a diameter of 19 mm. The chamber is filled with Argon gas to prevent oxidation of powder.

Rotation speed of the equipment is 3000rpm, which is measured with stroboscope, and the power is 40 KW. The powder is collected after the operation and is sieved, then mounted and polished. Powder of weight of 3g was then compacted in a mold of inner diameter of 12.50 mm and height of 5mm mixing together of 5% Zinc stearate.

The compacting pressure is 500 MPa and the sintering temperature is 1350 °C.

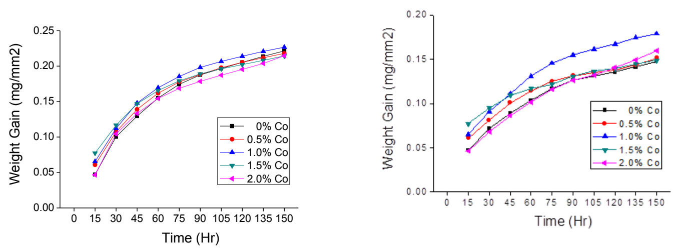
After oxidation test, the microstructure is observed by SEM, and the chemical components are analyzed by EDX.

The cyclic oxidation test is conducted with a heating rate of 10 °C/min to 850 °C with a holding time of 15 hours, then cooling and repeat heating to total time of 150 hours

Results and Discussion

Although the powder made of rotating electrode technique is clean in global shape, however the large particle size causes dense compacting difficult. In addition to this, composition of produced powders needed to be adjusted to the specification due to some vaporization loss. Hence different metallic fine powders, which include iron, chromium, cobalt and manganese powders were mixed with the global large size powder to improve the porosity and meet the specified composition. The amounts of fine powders used in this work were 40% and 60% both with particle size of 10 μ m.

In order to get a good oxidation resistance alloy, different quantities of cobalt were melted to form various composition alloys of Fe- 20Cr- 2Mn-X.



(a) 40% (b) 60%

Fig.1 the oxidation test of sintered powder mixing of (a)40% fine powder (b) 60%

From Fig.1 (a) and Fig.1 (b), it could be seen that 2.0% cobalt could have better oxidation resistance than others. The microstructure of exemplar sintered part of Fe- 20Cr- 2Mn-0.5% Co alloys after oxidation tests were observed with SEM, which is shown in Fig. 2. The results indicated that the microstructure is almost free from inclusion and contains very fine oxide surface. Fig.2 shows also that both 0.5 and 1.5% Co alloyed samples contain fine oxide.

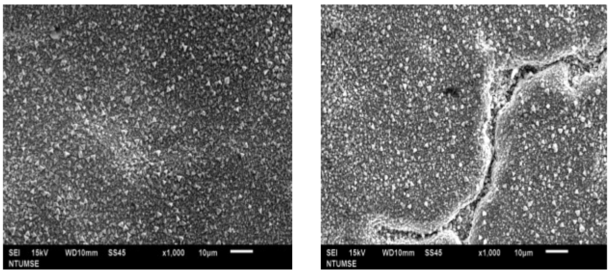


Fig.2 (a) Fe- 20Cr- 2Mn- 1.5 Co (b)Fe- 20Cr- 2Mn- 0.5 Co oxidation test at 850^{o}C 150 hours.

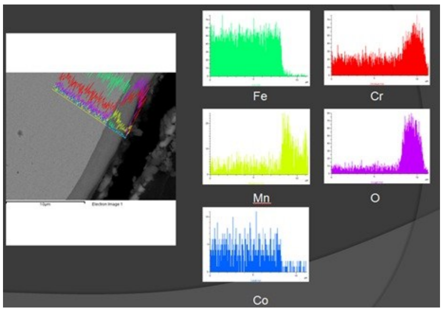
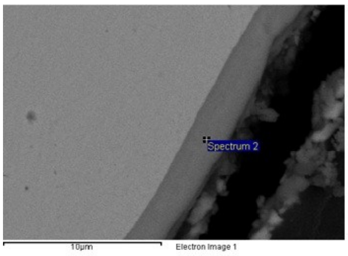


Fig.3 The line scans of oxide layer and inner part of substrate of oxidation samples.



Element	Weight%	Atomic%
O K	36.48	65.25
Cr K	57.06	31.40
Mn K	5.43	2.83
Fe K	0.83	0.43
Co K	0.19	0.09
Totals	100.00	

Fig.4 The analysis of oxide composition

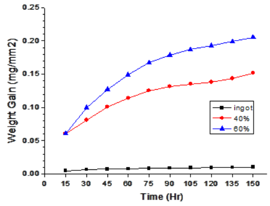
Fig.3 and Fig.4 indicate that the oxide layer adhere well on the substrate. The line scan (Fig.3) and EDX (Fig.4) denote that oxide layer is mainly composed of chromium oxide plus small part of manganese oxide or Cobalt oxide depending on the alloy elements.

The reason of cobalt oxide support the oxidation resistance of this alloy might be due to the formation of electron hole, which can retard the formation of oxygen ion of oxide layer.

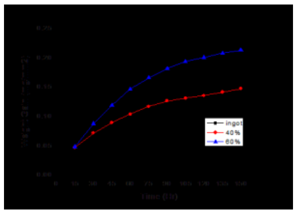
$$Cr^{2+} + Cr^{3+} = Co'_{Cr} + h \qquad O = O^{2-} + 2e$$

In order to compare the effect of fine powder addition, the sintered samples were conducted with the oxidation test without oxide coating. Sintered FeCr alloy powder and arc melted ingot are represented in Fig.5 (a)for 0.5%Co added to the alloy Fig.5 (b) for 0% added to the alloy powders. Both diagrams show that the sintered parts without coating of normal oxide layer have inferior oxidation resistance than normal wrought product due to the porosity.

However, the sintered samples could improve the oxidation resistance by proper addition of alloy element of cobalt with mixing of suitable amounts of fine powders. In this work, the addition of 60% of fine powder with 2.0% Co shows the best oxidation resistance.



5(a) Addition of 0.5%Co



5(b) Addition of 0%Co

Conclusion

1. The study has found that the sample made of rotating electrode powder is clean nearly free of inclusion.
2. The oxidation layer has good adhesion with the substrate.
3. The oxidation resistance at high temperature could be improved with addition of 60% fine powder and composition of Fe- 20Cr-2Mn-2% Co alloy

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[5]US Patent 6835227