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Contributed Paper

Physical Properties and Microstructure of a Fe-Ni-Co Alloy Prepared by Sintering Metal Powders Under Nitrogen Gas Atmosphere

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ABSTRACT

A route for preparing the Fe-Ni-Co alloy was studied. The mixed powders was compressed and sintered under nitrogen gas atmosphere. The mixing composition of metal powders was 54wt%Fe, 29wt%Ni and 17wt%Co. A single-action compression was applied together with a double press-double sinter (P_2S_2) process. Cooling of specimens was performed by quenching in ice water. Apparent density of 92% of the full density could be obtained. The microhardness of the prepared alloy was in the range of 110-144 HV. Details of microstructure compared with the commercial alloy were given.

Keywords : Fe-Ni-Co alloy, sintering, physical properties, microstructure

1. INTRODUCTION

In glass-to-metal joining, a metal alloy which has been used to join with the borosilicate glasses is the Fe-Ni-Co alloy because of the minimum thermal expansion mismatch [1-3]. The compositions of this glass-sealing alloy were in the range of 45-58wt%Fe, 28-32wt%Ni and 10-25wt%Co [1]. A ternary phase diagram with a liquidus projection has been reported as shown in Figure 1 [4]. The compositions of the glass-sealing Fe-Ni-Co alloy are within the vicinity where the dash lines crossover. The full density of the alloy was reported as 8.30 g/cm^3 [2]. Standard specification for iron-nickel-cobalt sealing alloy was given by ASTM F15-78 [5]. According to this ASTM designation, the alloy containing 54wt%Fe, 29wt%Ni and 17wt%Co should possess ASTM grain size less than No.5 and the hardness of 85 HRB when the thickness is more than 2.54 mm.

Production of the Fe-Ni-Co alloy can be done by casting following by calendering or extrusion. However, the potential process given near net shape is a production by powder metallurgy. Thornburg [2] succeeded in producing this alloy from metal powders by a double-sintering process under hydrogen gas atmosphere. However, use of the inert nitrogen gas could be another option making the process easier to control. Hence the purpose of the present work is to study the feasibility of preparing a Fe-Ni-Co alloy by sintering metal powders under the nitrogen gas atmosphere and to examine the physical preperties and microstructure of the alloy.

2. MATERIALS AND METHODS

2.1 Metal Powders

High purity Fe(>99.0wt%), Ni(99.8wt%)

and Co(99.8wt%) powders were used. The particle size is less than 325 mesh for all powders. Morphology of them was studied by scanning electron microscopy (SEM) using JEOL 840A scanning electron microscope.

2.2 Compression

A compression die as shown in Figure 2a was made from tool steel D2, which was surface-hardened for obtaining a surface hardness of 55 HRC. Dimensions of the die was designed following ASTM B331-85 [6]. Metal powders weighted as 54wt%Fe, 29wt%Ni and 17wt%Co were mixed in a rotary mixer for 5 hours. Portions of 10.00 g of the mixed powders were transfered into the die. A uniaxial single-action compression was applied as shown in Figure 2b. Initially, the compacting pressure of 456 MN/m^2 (30 tons/in^2) was applied for 2 minutes. The green specimens obtained were taken out from the die and pre-sintered in a tube furnace at 950 oC for 2 hours under the atmosphere of the nitrogen gas, controlling the flow rate as 8.33×10^{-5} m^3/s (5 l/min). The specimens were cooled within the furnace. The pre-sintered specimens were then put back into the die and the second compression was performed using the pressure in the range of 532-760 MN/m^2 (35-50 tons/in^2) for 2 minutes. Finally, the specimens were sintered again in the furnace under the same condition as for pre-sintering, except that cooling was performed by quenching in ice water.

2.3 Physical Properties Measurement

Green density of compressed specimens was calculated directly from their dimensions after compression, whereas the density of the sintered specimens was measured by buoyancy force method. Vickers microhardness of the sintered alloy was measured using Galileo Microscan OD system at 500 gf load and 15 s indenting time.

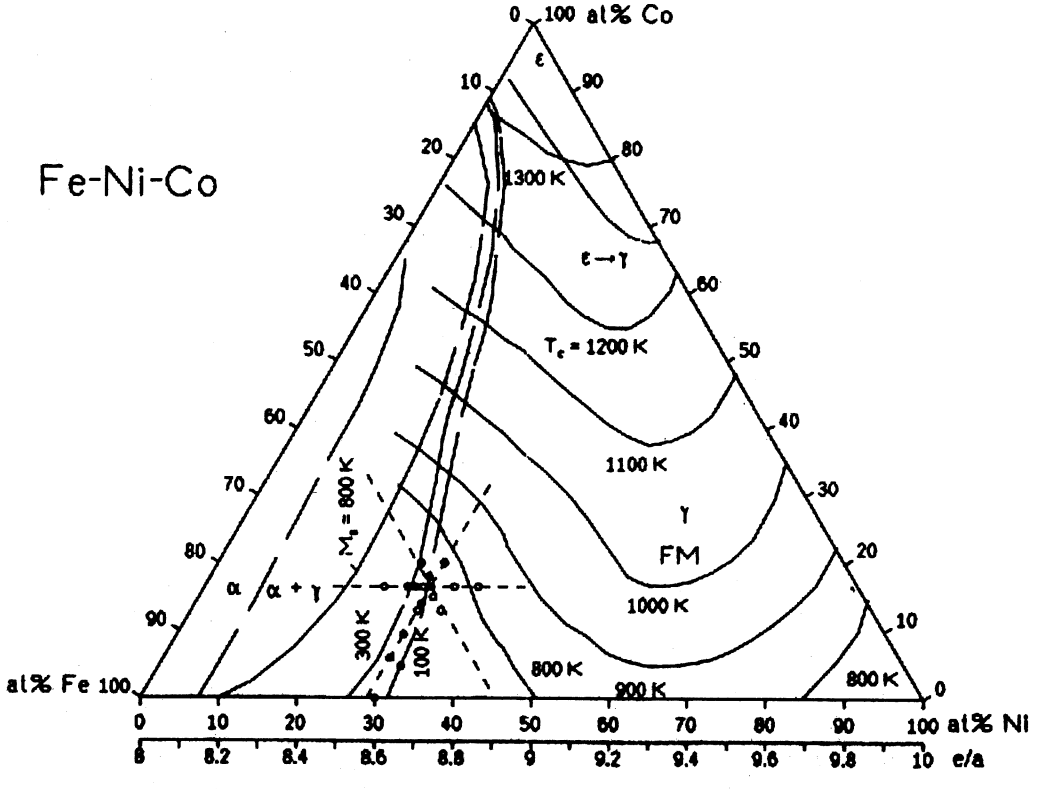


Figure 1. A ternary phase diagram with liquidus projection of Fe-Ni-Co system [4].

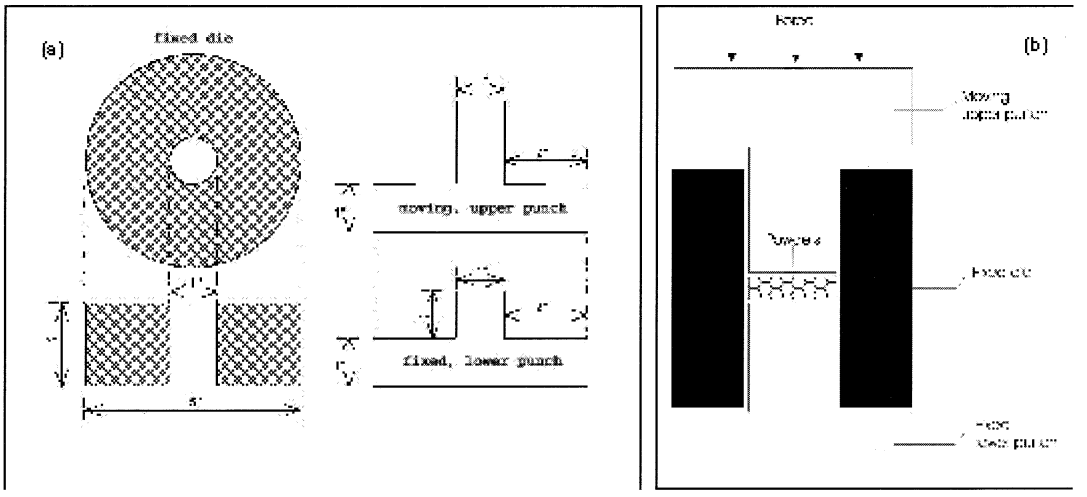


Figure 2. (a) Dimensions of the compression die, (b) Single-action compression.

2.4 Microstructural Examination

Polished specimens were chemically etched using a solution prepared from 1 part by volume of concentrated nitric acid satura ted with $CuCl_2.2H_2O$ and 3 part by volume of concentrated hydrochloric acid. Light microscopy was performed using Olympus BX60M microscope. Grain size was measured from micrographs by linear intercepts in several directions. Porosity was determined by point counting method. Chemical analysis of alloy composition were studied by scanning electron microscopy-energy dispersive x-ray spectrometry (SEM-EDS). Crytal structure was also examined by x-ray diffraction (XRD) using Bruker AXS D8 diffractometer.

3. Results and Discussion

3.1 Morphology of Metal Powders

Figures 3a-3c show morphology of metal powders used in this work. It can be seen that the iron and cobalt powders are more or less spherical particles. The surface of the iron particles is faceted but that of the cobalt particles is smooth. On the other hand, nickel particles are somewhat agglomerated ligament. The particle size of these powders measured from the micrographs was less than 8 microns.

3.2 Physical Properties Measurement

The green density of the specimens before sintering was 6.46±0.25 g/cm^3. After a double press-double sinter (P_2S_2) process and quenching, the density of the specimens was increased to be 7.66±0.50 g/cm^3. This is equivalent to about 92% of the full density (8.30 g/cm^3). It was found that the effect of the force in the second compression, i.e. 35-50 tons/in^2, to the density of the sintered specimens was negligible. The microhardness of the double-sintered specimens was in the range of 110-144 HV. The effect of the force in the second compression on the microhardness is shown in Figure 4. It can be seen that the microhardness tent to decrease slightly with the increase of the compression force.

3.3 Microstructural Examination

Figure 5 exemplifies a typical microstructure of the alloy prepared in this work. Grains of a single phase were observed with twin boundaries. This could be the f.c.c g phase according to the phase diagram given by Gehrmann *et al.* [4]. The grain size of specimens recompressed at different forces was in the range of 17-22 microns as shown in Figure 6, increasing with the increase of the compression force. It is possible that the more plastic deform enhanced grain growth during sintering and hence the microhardness of the specimen recompressed at higher force was slightly lower. Pores were also observed and distri-

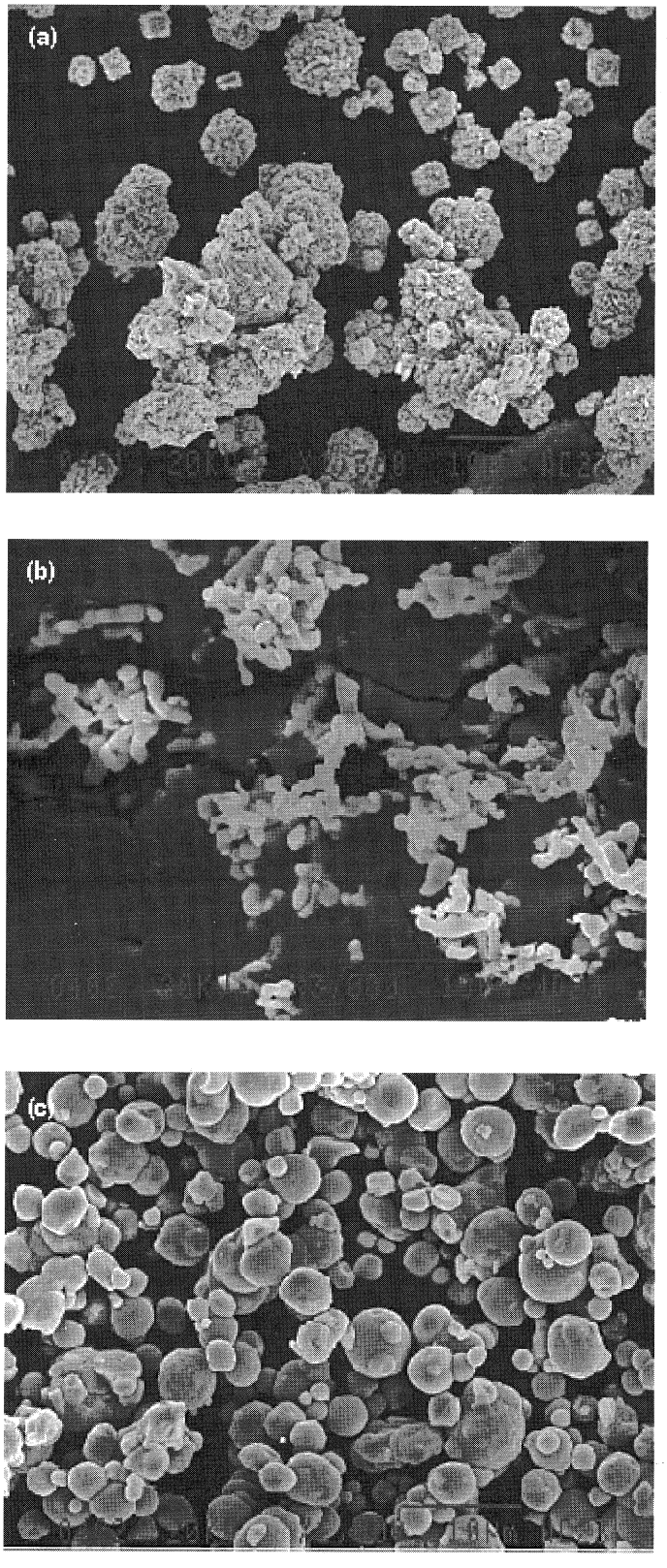


Figure 3. Secondary electron images of metal powders from SEM. (a) Fe, (b) Ni and (c) Co.

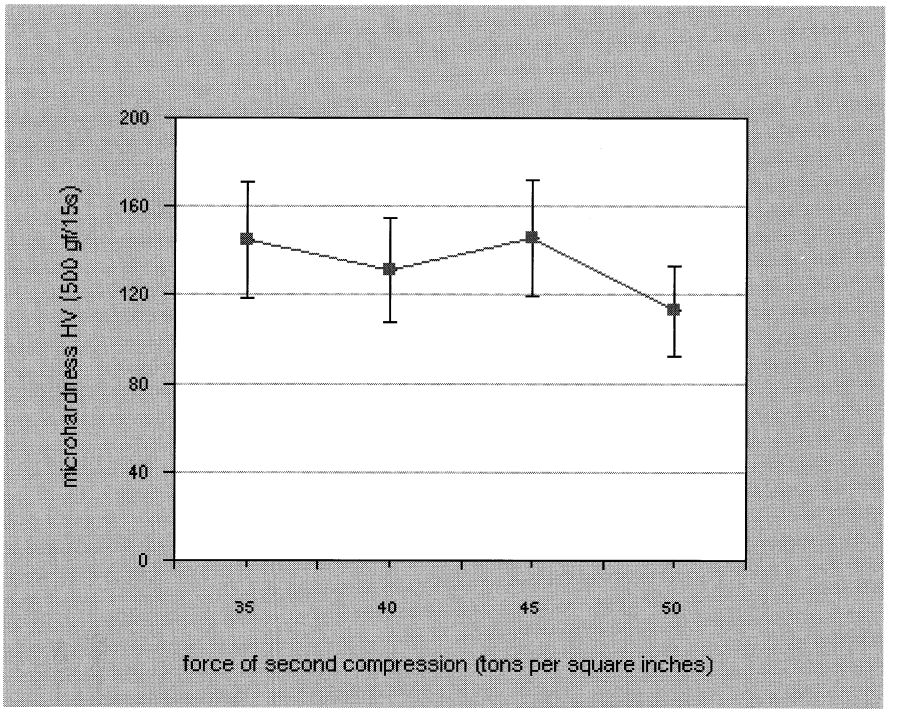


Figure 4. The effect of the force in the second compression on the microhardness of the specimens after sintering and quenching.

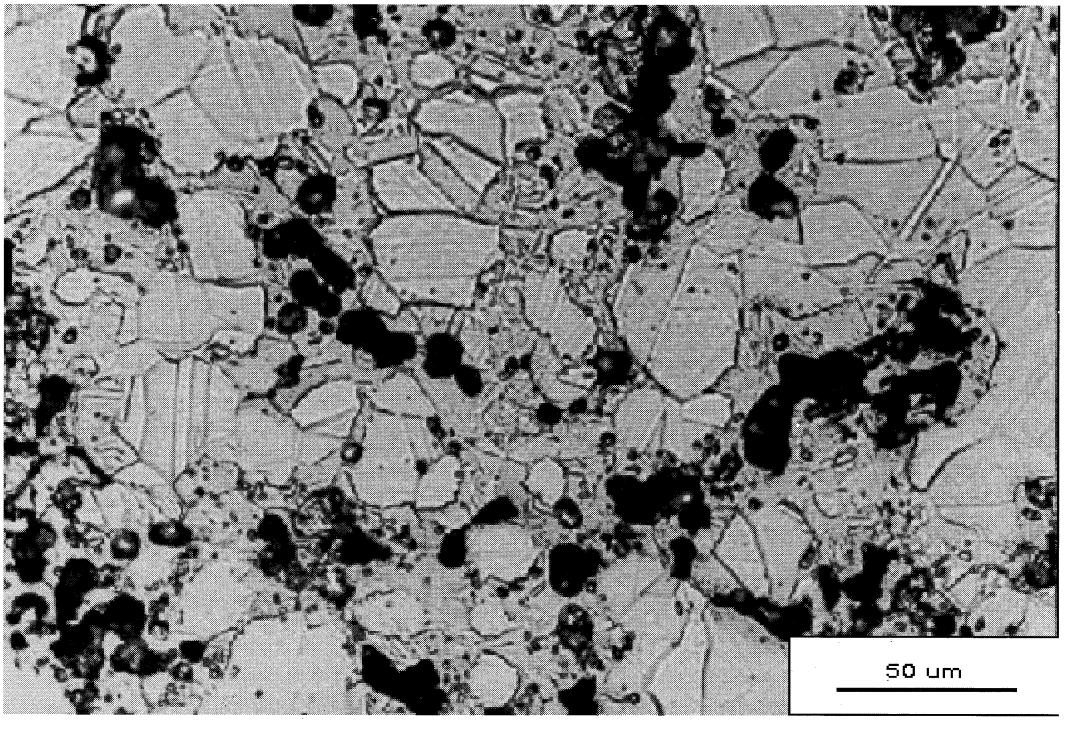


Figure 5. A light microscopy showing a typical microstructure of the Fe-Ni-Co alloy prepared in this experiment.

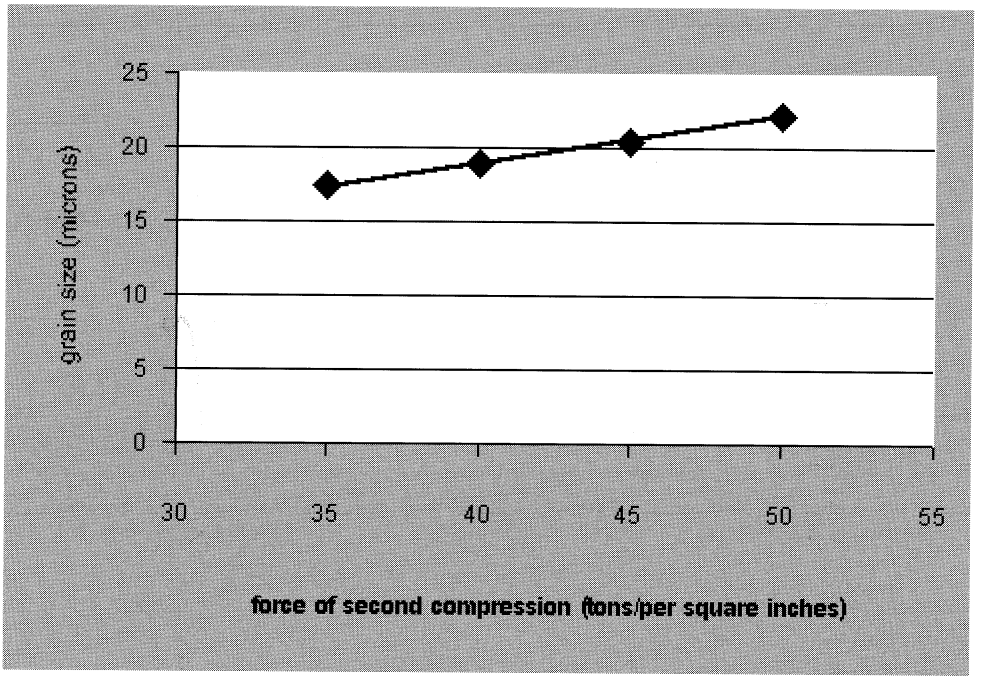


Figure 6. The effect of the force in the second compression on the grain size of the specimens after sintering and quenching.

buted randomly in the bulk. The porosity measured by point counting was about 1-7%. These pores are undesirable and further study is needed to improve the process for getting the full density.

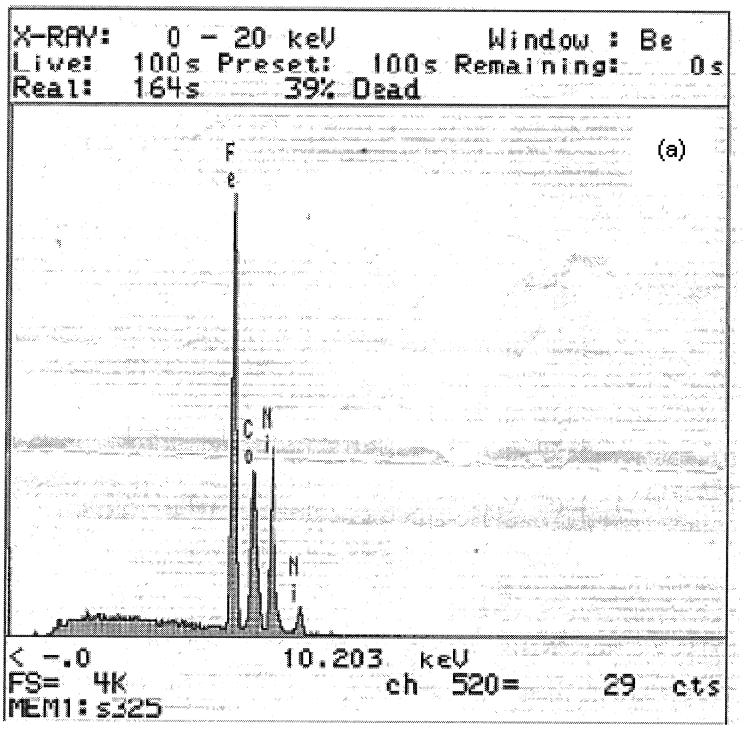
The result from SEM-EDS study of all specimens were consistent and a spectrum was given in Figure 7a. The relative content of the iron, nickel and cobalt corresponded to the mixing ratio of the metal powders. The XRD spectrum of the alloy was shown in Figure 7b, where two strong peaks were found at 2θ = 44.2 and 51.4 degree. Matching with JCPDS database, the structure could be of Awaruite type containing Co. This is comparable to the microstructure, the chemical composition and the crystal structure of a commercial Fe-Ni-Co alloy shown in Figure 8. The reason of the different relative intensities of the two peaks in XRD spectrums could be attributed to the different in grain orientation or texture of the two alloys.

4. Conclusions

4.1 Preparation of the Fe-Ni-Co alloy by sintering of metal powders under the nitrogen gas atmosphere can be another processing option.

4.2 The alloy could be prepared by a double press-double sinter (P_2S_2) process followed by quenching in ice water. This alloy possessed a density of 7.66±0.50 g/cm3, the microhardness of 110-144 HV and the grain size 17-22 microns. The microstructure of the prepared alloy was comparable to a commercial alloy.

4.3 Porosity was still remained in the bulk. This requires a further study to obtain the full density.



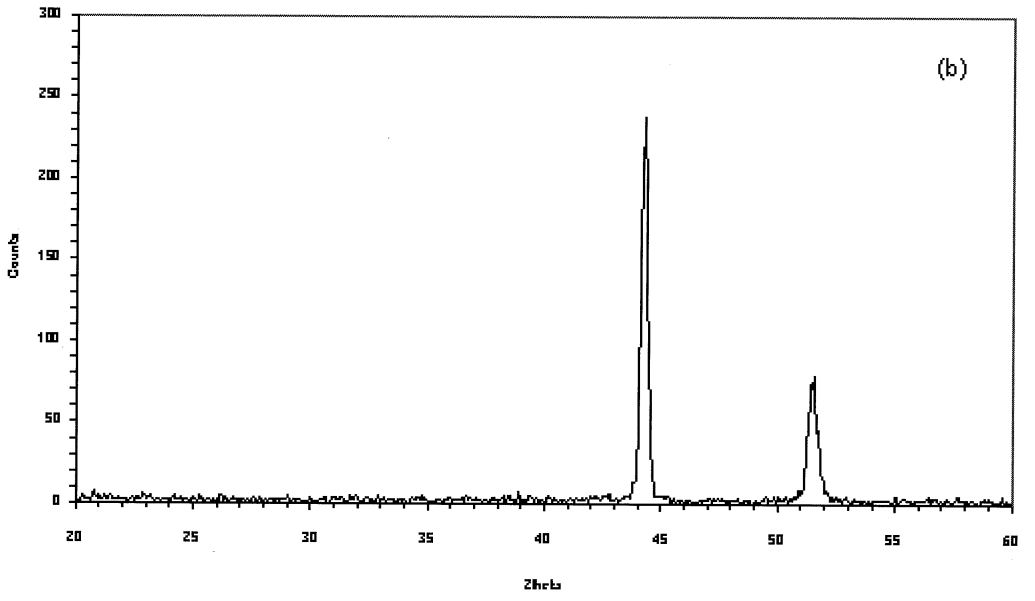


Figure 7. (a) SEM-EDS spectrum and (b) XRD spectrum of the alloy prepared in this experiment.

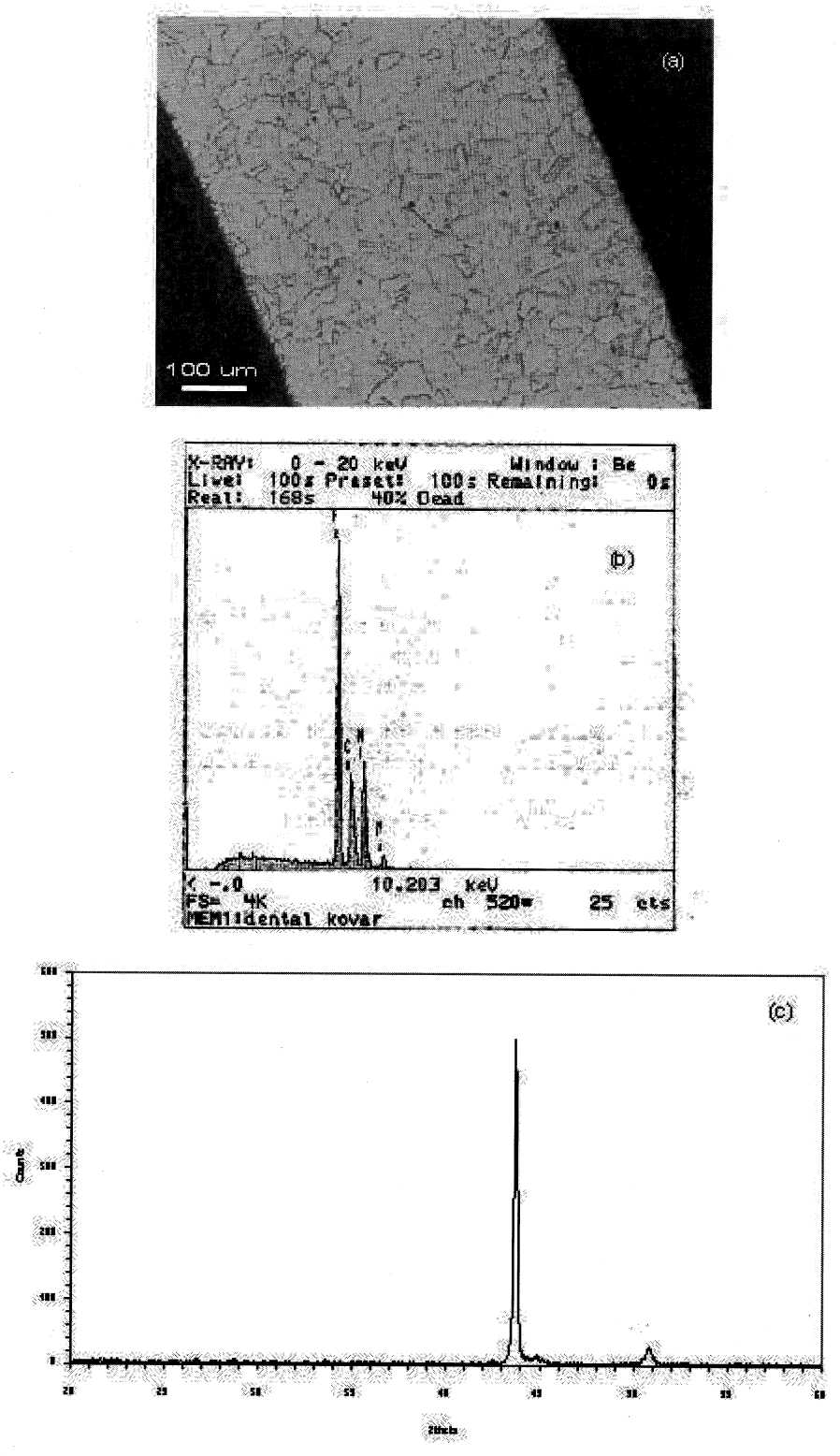


Figure 8. A commercial Fe-Ni-Co alloy. (a) A light microscopy showing the microstructure, (b) SEM-EDS spectrum and (c) XRD spectrum.

ACKNOWLEDGEMENTS

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