

Amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ Powder with High Corrosion Resistance

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Amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ powder has been prepared by the spark erosion technique and its corrosion behavior investigated potentiodynamically. It is concluded that the powder prepared this way possesses a relatively high corrosion resistance, as does amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ ribbon prepared by rapid quenching.

Introduction

Since the successful work of Duwez [1] many investigations on preparation techniques, properties and practical applications of amorphous alloys have been performed. Except for thin films, the preparation techniques depended mainly on splat-cooling, because the ribbon-shaped products obtained by this method are useful for fundamental measurements and applications. However, it is still of interest from theoretical and practical points of view to advance new techniques for preparing amorphous alloys.

The spark erosion was employed by Bucklow and Drain [2] and Ruppertsburg and Bold [3] to obtain very fine powders of some metallic elements and alloys. Anantharaman [4], Filimonenko et al. [5] and Krainer and Robitsch [6] have discussed the metastable crystalline phases of WC-Co and Co formed on the surface of spark discharged electrodes. Recently, Yamaguchi and Narita [7] have successfully prepared transition metal-metalloid amorphous powders by the spark erosion technique. On the other hand, Naka, Hashimoto and Masumoto [8] have reported that amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ ribbon prepared by the rapid quenching method reveals an extremely high corrosion resistance in some solutions.

In the present work amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ powder with high corrosion resistance has been prepared by means of electro-spark erosion.

Experimental

All ingots used were prepared by melting commercial grade electrolytic iron (99.9%), metallic

chromium (99.9%), ferro-phosphorus (26.8% P) and carbon in a high frequency induction furnace under an argon atmosphere. The casted ingots were cut and polished mechanically to be used as positive and the negative electrodes in the spark erosion process carried out by an ordinary electrodischarge machining apparatus (EKOMIX DE75-3CF), using kerosene as the dielectric fluid. The discharge voltage was 100 V. The powder was filtrated and washed with acetone to remove the kerosene. The powder was found by x-ray diffraction to be amorphous, and its density, Vickers hardness and crystallization temperature were measured.

From the amorphous powder (grain size below $20\mu\text{m}$) pellets were made with a pressure of $500\text{ kg}\cdot\text{cm}^{-2}$. Their corrosion was studied in 1 N-HCl, 1 N- H_2SO_4 , 1 N-NaOH, 3% NaCl and 10% $\text{FeCl}_3\cdot 6\text{H}_2\text{O}$, which were prepared with reagent grade chemicals and deionized water. Prior to the electrochemical measurements, the pellets were polished with silicon carbide paper of grades up to No. 1000. The electrolytes employed were exposed to air. Anodic polarization curves were measured potentiodynamically with a potential sweep rate of $4\cdot 10^2\text{ sec}\cdot\text{V}^{-1}$, starting from the corrosion potential at room temperature. A new specimen and a new solution were used for each potentiodynamic polarization measurement.

Results

Figure 1 shows the x-ray diffraction pattern of the powder obtained by the spark erosion, which indicates a typical halo pattern of an amorphous state. Figure 2 shows a scanning electron micrograph (SEM) of the amorphous powder, which consists of roughly spherical particles with diameter of 1 to $20\mu\text{m}$.

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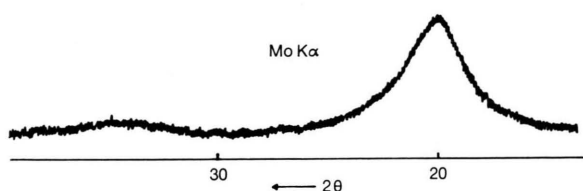


Fig. 1. X-ray Diffraction Pattern of $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ Powder Prepared by Spark Erosion.

The differential scanning calorimetric measurement of the powder revealed one stage of crystallization from which the crystallization temperature (T_c) of the amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ powder was estimated to be 790.5 K at the heating rate of $20 \text{ deg} \cdot \text{min}^{-1}$. On the other hand, T_c of the amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ ribbon prepared by the rapid quenching method has been reported to be 709 K by the authors in [9]. The difference seems to be caused by the difference of the crystallization mechanism rather than experimental errors. This point will be discussed later.

The density by pycnometer of the amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ pellets is $2.97 \pm 0.11 \text{ gr} \cdot \text{cm}^{-3}$ and the Vickers hardness by micro-Vickers is 135 under 100 gr of load.

Weight changes of the specimens in the solutions were impossible to estimate because the solutions penetrated into the pellets. Examination with the naked eye showed little corrosion, if any. Figure 3 shows the natural potential change of the amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ powder in the solutions. After the specimen anodes were immersed in the solutions, the natural potentials changed immediately to become almost constant during an hour. The steady

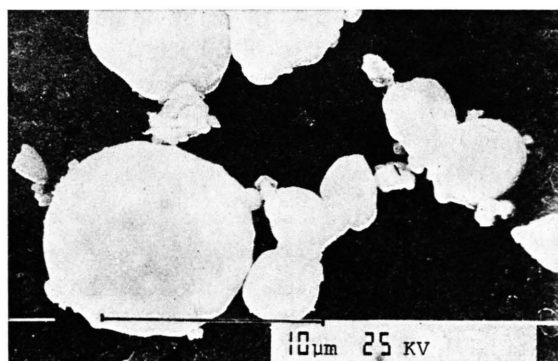


Fig. 2. Scanning Electron Micrograph of $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ Amorphous Powder Prepared by Spark Erosion.

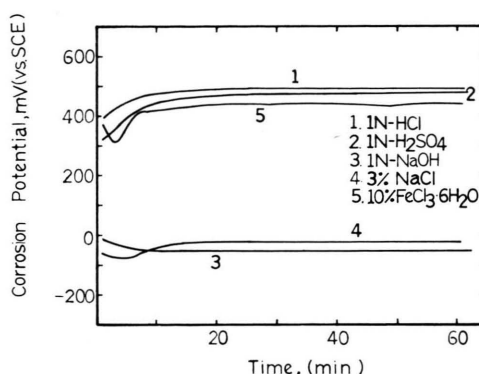


Fig. 3. Natural Potential Change of Amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ Powder.

state potentials were in the region of passive potentials.

Figure 4 shows the anodic polarization curves. The polarization curves for the amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ ribbon reported by Naka *et al.* [8] are included in the figure. They show similar trends. It seems that the amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ powder prepared by the erosion technique has a considerably higher corrosion resistance than the amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ ribbon. As also seen in Fig. 4, the passive regions of the powder are wider than those of the ribbon, which is supposed to be due to a fairly firm passive thin film.

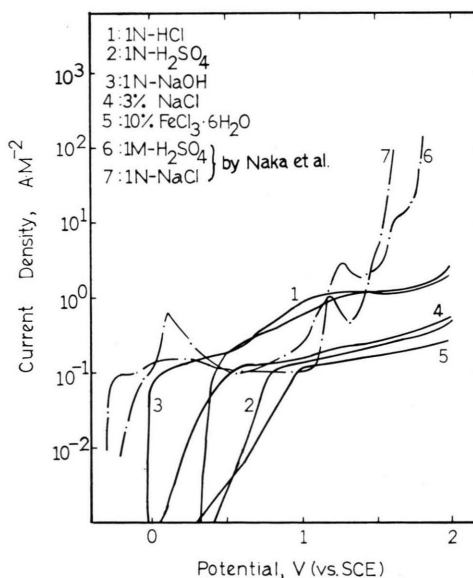


Fig. 4. Anodic Polarization Curves of Amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ Powder.

Discussion

The amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ powder can be successfully prepared by the spark erosion technique if the same original master ingots are used for both electrodes. However, if a copper plate was used as the cathode, amorphous powder containing crystalline copper was produced. The amount of copper was analyzed after magnetic separation by atomic absorption measurements to be about 10 wt.%.

Vaporization or fusion may be involved in the formation mechanism of the particles by spark erosion [10, 11]. Yamaguchi *et al.* [6] have suggested from their SEM studies that the latter mechanism would be more dominant because of the presence of craters. In the present work, however, no craters could be found in the SEM observation. Hence it is not certain which mechanism was dominant.

The higher crystallization temperature of the amorphous powder prepared by the spark erosion as compared to that of the rapidly quenched amorphous ribbon seems to suggest that the amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ powder has a better thermal stability than the amorphous ribbon.

The powder prepared with a copper cathode showed a poorer corrosion resistance than the powder containing crystalline copper. The anodic polariza-

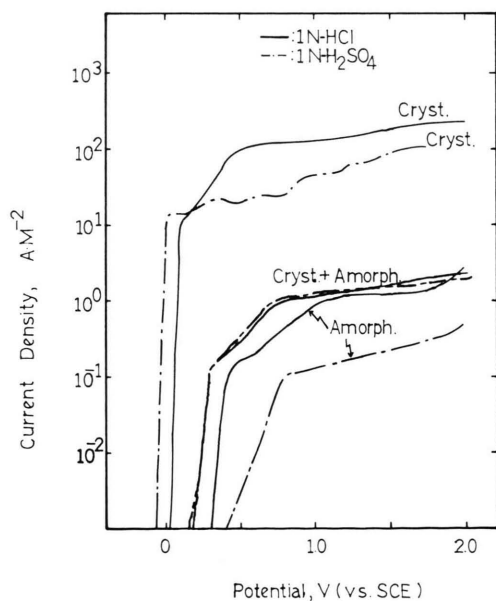


Fig. 5. Comparison of Anodic Polarization Curves of Crystalline and Amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ in Acidic Solutions.

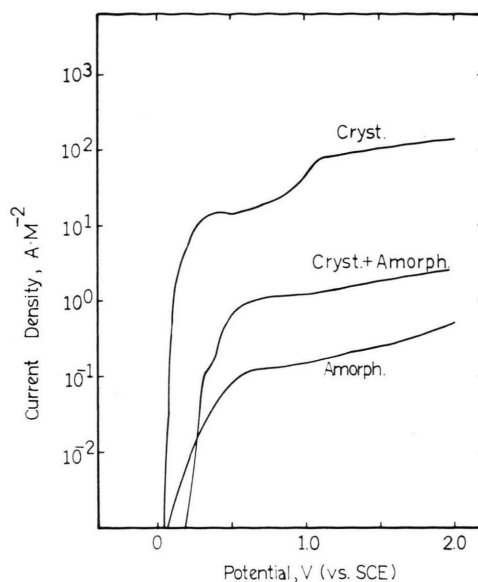


Fig. 6. Comparison of Anodic Polarization Curves of Crystalline Amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ Powder in 3% NaCl Solution.

tion curves for the amorphous and crystalline powders in 1 N-HCl, 1 N- H_2SO_4 and 3% NaCl solutions are shown in Figures 5 and 6. As seen in the figures, the amorphous powder containing crystalline copper shows higher corrosion resistance than that of the corresponding crystalline alloys and poorer corrosion resistance than that of the amorphous powder without crystalline copper.

Conclusion

In conclusion, the following statements can be made: The amorphous $\text{Fe}_{72}\text{Cr}_8\text{P}_{13}\text{C}_7$ powder can be prepared by the spark erosion technique and reveals a relatively high corrosion resistance, as does the amorphous ribbon prepared by the rapid quenching method. The amorphous powder by this technique differs in some features from the splat-cooled amorphous ribbons, especially as to the crystallization temperature. It is expected that these results help to develop new materials with high corrosion resistance.

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