

the designers [3] of Dispersalloy have, perhaps inadvertently, optimized the performance of the additive particles to give a suitable balance between the first stage precipitation of η' and the second stage gettering of Sn.

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Vickers micro-hardness of solid solution in the system $\text{Cr}_2\text{O}_3\text{--Al}_2\text{O}_3$

Aluminium oxide and chromium oxide form a continuous solid solution above 1000°C [1], and materials with a wide variety of properties are expected. In this communication, the Vickers micro-hardness was measured for the system $\text{Cr}_2\text{O}_3\text{--Al}_2\text{O}_3$ containing Cr_2O_3 up to 50 mol%.

Aluminium ammonium alum (Wako Junyaku, Reagent grade), chromium ammonium alum (Kanto Kagaku, Reagent grade) and magnesium sulphate (Wako Junyaku, Reagent grade) were used as starting materials. The specimens prepared are given in Table I. All specimens contained 0.7 mol% MgO as grain growth inhibitor. Weighed starting materials were mixed and heated in an

alumina crucible at 550°C for 5 h, 950°C for 15 h and 1200°C for 3 h. The resultant oxide was pulverized for 1 h in an alumina ball mill. The formation of solid solution was confirmed by powder X-ray diffraction analysis ($\text{CuK}\alpha_1$, APD-10, Philips); the lattice parameters followed the Vegard's law. Rectangular bars (4 mm \times 2 mm \times 50 mm) were formed from the oxide powders at a pressure of 250 kg cm^{-2} and pressed isostatically at 1000 kg cm^{-2} . They were buried in oxide powder of the same composition and sintered at 1600°C for 5 h in a water-saturated hydrogen stream or at 1700°C for 5 h in air. The apparent densities of sintered specimens were determined from their weights and dimensions. The specimens were buried in resin and their surfaces were polished with $\frac{1}{2}\mu\text{m}$ diamond paste. The Vickers

TABLE I Characteristics of specimens

Starting oxide		Sintered material 1600° C, 5 h in H ₂ + H ₂ O		Sintered material 1700° C, 5 h in air		
Composition Cr ₂ O ₃ (mol %)	Lattice parameters (Å)		Apparent density (g cm ⁻³)	Lattice parameters (Å)		Apparent density (g cm ⁻³)
	a	c		a	c	
0	4.7590	12.991	3.86	4.7590	12.993	3.84
5	4.7697	12.018	3.97	4.7704	13.023	3.95
10	4.7798	13.043	3.94	4.7837	13.057	4.01
20	4.8028	13.107	4.05	4.8059	13.116	4.05
50	4.8651	13.293	4.47	4.8679	13.293	3.78

micro-hardness was measured with the applied load 200 g at the room temperature. The characteristics of specimens are given in Table I.

Fig. 1 shows the Vickers micro-hardness of sintered specimens. The hardness could be easily determined on all specimens sintered in water-saturated hydrogen, and those with a chromium content of 0 to 20 mol% sintered in air. The specimen with a chromium content of 50 mol% was very porous when sintered in air (see Table I), and its hardness could not be determined. Essentially, same results were obtained on the specimens sintered in air and in water-saturated hydrogen. The hardness increased sharply with increasing

chromium content, reached a maximum at approximately 20 mol% and decreased at higher chromium content. The increase in hardness with increasing chromium content agreed with the results of Ghate *et al.* who prepared the specimens by hot-pressing [2]. The concentration which gave the maximum hardness was, however, different; 20 mol% in the present study and 10 mol% in the study by Ghate *et al.* The hardness was slightly higher in the present study at the same composition. The lighter load (200 g versus 500 g) is mainly responsible for the difference. The hardness of specimens containing no chromium agreed with the literature value [3].

The slightly lower hardness of specimens sintered in air may be due to the evaporation of chromium from the surface region. The vapour pressure of chromium oxide is much higher in air than in water-saturated hydrogen [4]. The higher sintering temperature in air than in water-saturated hydrogen is also favourable for the evaporation loss of chromium. Decrease in chromium content from the surface region reduced the Vickers micro-hardness. In addition to this, microstructure and non-stoichiometry may also be responsible for the slight difference in hardness in the region of high chromium content.

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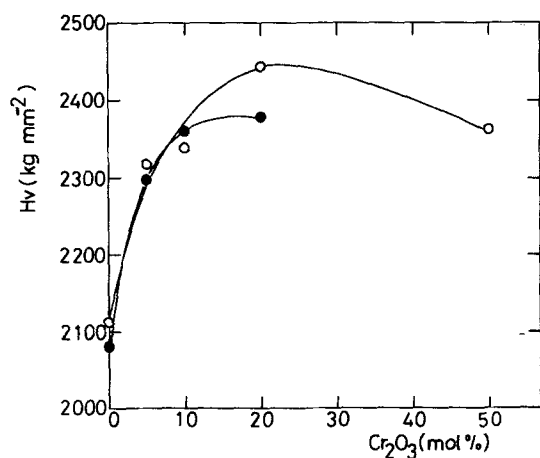


Figure 1 Effect of Cr₂O₃ content on the Vickers micro-hardness in the system Cr₂O₃-Al₂O₃. ○: specimens sintered at 1600° C for 5 h in H₂ + H₂O. ●: specimens sintered at 1700° C for 5 h in air.

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Electrolytic durability of glassy carbon

Graphite carbon is widely used as a cheap and relatively durable electrode material for batteries and in the processes of electrolytic industry [1]. Glassy carbon (GC), a special amorphous carbon modification [2], prepared by slow pyrolysis of certain polymer materials, has proved to be suitable for many applications in electroanalytical chemistry [3] and other electrochemical studies [4]. To elucidate the suitability of GC for electrodes in electrolysis we performed this study where the durabilities of various GC and graphite electrodes were compared in a prolonged electrolysis of aqueous solutions.

Round rods of GC (diameter 8 mm) were prepared with a pyrolytic method described earlier [5]. Three different carbonization temperatures, 800, 900 and 1000°C, were used. The electrical resistivities of the products were 3.9, 0.75, and 0.26 mΩ m, respectively, which correspond well with the values reported in the literature [2]. GC pieces of this size are porous. During 1 month's soaking in distilled water, the weights of a few sample pieces increased by 18 to 31%. The pores were, however, very small; their diameters were about 30 nm as measured from SEM photographs. Non-porous GC electrodes were made by cutting rectangular rods with a diamond saw from commercial GC discs (manufactured by Atomergic Chemetals Corp, New York, USA, and an unknown manufacturer in USSR). The graphite electrodes were synthetic battery electrodes, 8 mm × 57 mm, manufactured by Dae Han Carbon Co, Ltd, Korea.

In the electrolytic runs 1 N hydrochloric acid, sulphuric acid, sodium chloride, and sodium hydroxide solutions prepared from analytical

grade reagents were used. Sixteen plastic electrolytic vessels were connected in series and a voltage of 2.8 V was applied between each electrode pair. This caused the evolution of hydrogen, oxygen and chlorine gasses and produced a steady state current of 30 mA (about 10 A m⁻²). At proper intervals the electrodes were removed, washed, dried, and weighed. At the same time the solutions were replaced with fresh ones.

The results of our experiments are presented in Table I. The carbon electrodes exhibit good cathodic durability except for the porous GC electrodes in the solutions containing sodium ions. They then became brittle and crumbled either in the solutions or during the drying before weighing. This interesting phenomenon resembles the attack (intercalation) of gaseous alkali metals [2] which even causes explosions of samples. The effect may now be due to the sodium metal reduced in the pores of the samples. As anodes, the carbon electrodes were poor, regardless of

TABLE I Electrolytic durabilities of various carbon electrodes

Electrode material	Solution, electrode and evolved gas							
	HCl		H ₂ SO ₄		NaCl		NaOH	
	H ₂	Cl ₂	H ₂	O ₂	H ₂	Cl ₂	H ₂	O ₂
GC (800° C)	G	B	G	B	M	B	B	B
GC (900° C)	G	B	G	B	B	B	B	B
GC (1000° C)	G	B	G	B	B	B	B	B
GC non-porous	G	B	G	B	G	M	G	B
Graphite	G	B	G	B	G	M	G	M

The durabilities are marked with the following notations:
 B = bad: significant weight loss (over 2%) in 50 h electrolysis;

M = medium: significant weight loss in 500 h electrolysis;
 G = good: no significant weight loss in 500h electrolysis.