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Distribution of elements in Ni–Mo–TiC alloy processed under microgravity conditions

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Ni–Mo–TiC particle-strengthened alloy was processed through melting–pressing treatment of hot-pressed samples under microgravity environments by sounding rocket flight. The distributions of titanium carbide particles and elements in the alloy sample obtained were analysed using microscopy and micro Auger electron spectroscopy. The objective of this analysis was mainly to clarify the elemental distribution in the titanium carbide particle. Analysis was also carried out on the melting–pressing treated and hot-pressed samples for comparison. Analytical results showed that titanium carbide particles are uniformly distributed by the processing under microgravity and that stable mutual diffusion reaction had progressed there.

1. Introduction

If fine particles of titanium carbide ceramics are uniformly dispersed in a nickel alloy matrix, excellent heat-resisting materials will be prepared. However, such materials have never been manufactured by the conventional melting and solidifying method which is the usual method used for alloy preparation.

The density of titanium carbide is 4.9, which is relatively small compared with other metals such as nickel and molybdenum, 8.9 and 10.2, respectively. Consequently, titanium carbide particles float to the surface of a molten alloy on the ground.

Thermal convection also has an influence. These phenomena can be neglected under microgravity conditions. Therefore, nickel alloy composite material with uniformly dispersed titanium carbide particles can be prepared by the melting–solidifying technique in such conditions. Nickel alloy strengthened by the dispersion of titanium carbide particles was prepared under microgravity conditions by the ballistic flight of a sounding rocket. This paper reports a study of the elemental distribution and structure of the sample obtained by the microgravity experiment.

2. Materials and methods

2.1. Samples

Three kinds of sample, HP, GM and FM, were studied. Sample HP was prepared by blending powders of 1 to 2 μm average diameter in the composition of 24% titanium carbide, 7% molybdenum, 3% carbon and the balance nickel, in atomic percentage. The blended powders were pressed under about 200 MPa, and then hot pressed into cylindrical form, 10 mm diameter and 35 mm long, at about 1260° C in a high-vacuum furnace.

Sample GM was prepared by pressing sample HP in the molten state at a pressure of 2 MPa in a terrestrial electric furnace which was made for simulation of the rocket flight experiment.

Sample FM was prepared by pressing sample HP in the molten state at a pressure of 2 MPa in an electrical furnace under microgravity conditions of the order of $10^{-4}g$ where g is the acceleration due to gravity, for about 6 min on the sounding rocket in flight. In this case, sample FM was rapidly heated to the molten state at about 1450° C within 2 min, kept at that temperature for 100 sec, pressed by a piston mechanism and then quickly cooled by blowing with helium gas.

Sample GM was prepared on the ground, by a process similar to that of sample FM, except for the microgravity conditions.

Fig. 1 shows the temperature–time curve measured during the microgravity experiment at a point near the sample in the graphite crucible [1]. Fig. 2 shows the appearance of sample FM obtained through the flight experiment and microstructures of samples FM and GM [1].

2.2. Experimental procedure

The sample was cut into round slices in order to observe the distribution of titanium carbide particles and to analyse the constituent elements of the samples.

From the microstructures, a non-uniform distribution of titanium carbide particles was observed only in sample GM. Because sample GM was melted and solidified in the vertical column as shown in Fig. 1, titanium carbide, whose density was smaller than those of other elements, became rich in the upper part of sample GM; thermal convection is also supposed to

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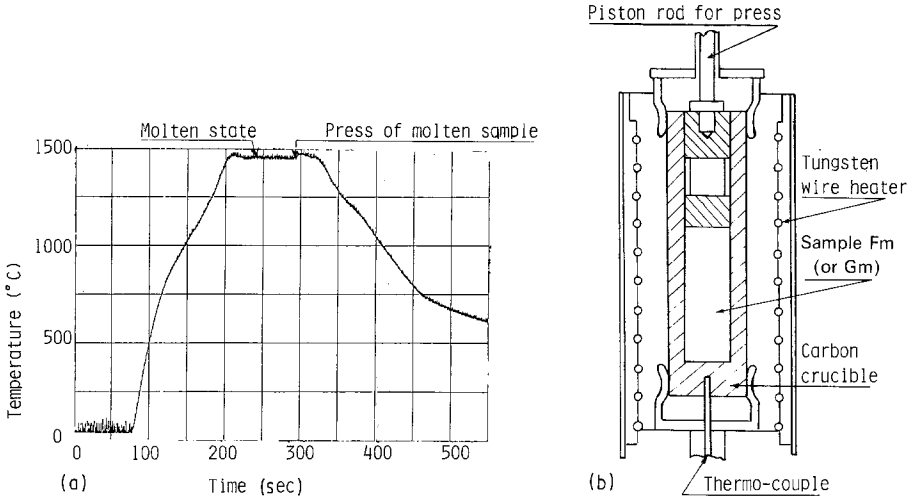


Figure 1 Temperature–time curve of electric furnace during the rocket flight experiment (a) and the sample in the graphite crucible (b) [1].

have contributed to the uneven distribution of titanium carbide particles.

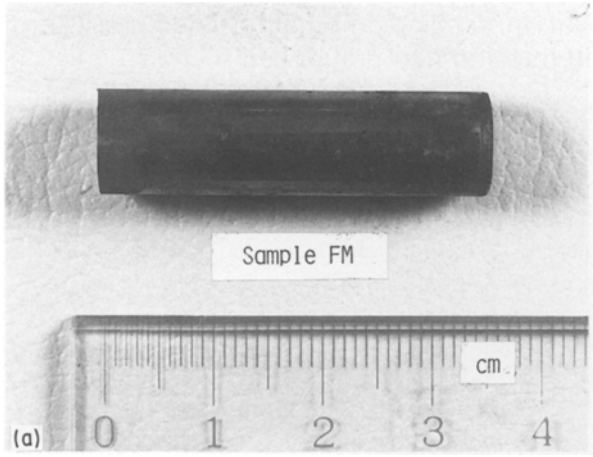
The polished surface of a sample was observed using a photomicroscope and a scanning electron microscope (SEM). After removal of carbon and oxygen on the surface of the polished samples by xenon ion sputtering treatment, 90 points of micro Auger electron spectroscopy (AES) measurements were performed for sample HP and 150 points for each sample GM and FM.

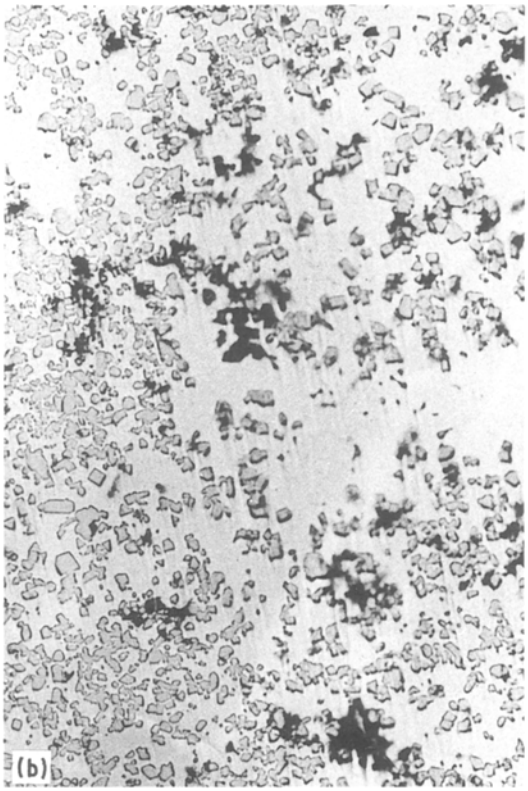
The distributions of elemental nickel, molybdenum, titanium and carbon were obtained by quantitative and statistical treatments of the measured data. The ambient pressure during AES measurement of the sample was about 1×10^{-7} Pa, and the diameter of the electron beam was about 0.3 μm.

3. Results and discussion

3.1. Microscopic structure

Fig. 2 shows an optical micrograph of the structure in cross-sections of samples GM and FM. The





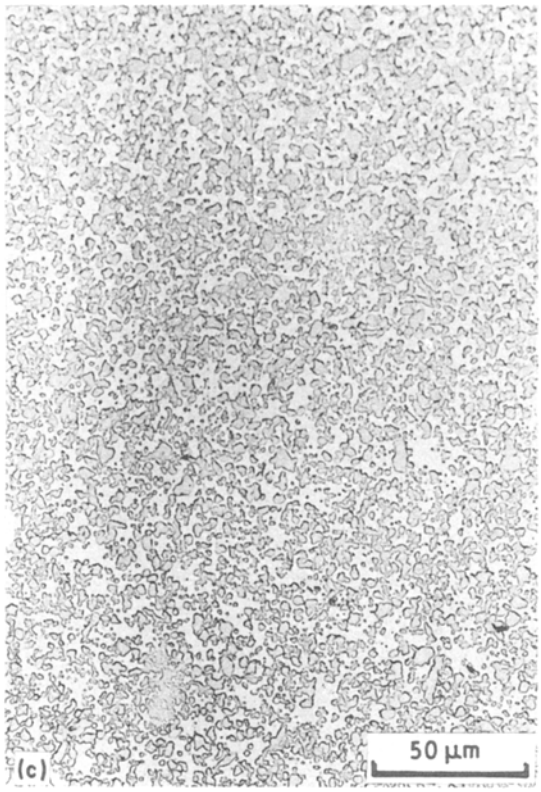


Figure 2 (a) Appearance of sample FM obtained through the rocket flight experiment, and the microstructures of samples (b) GM and (c) FM [1]. Sample GM was prepared on the ground.

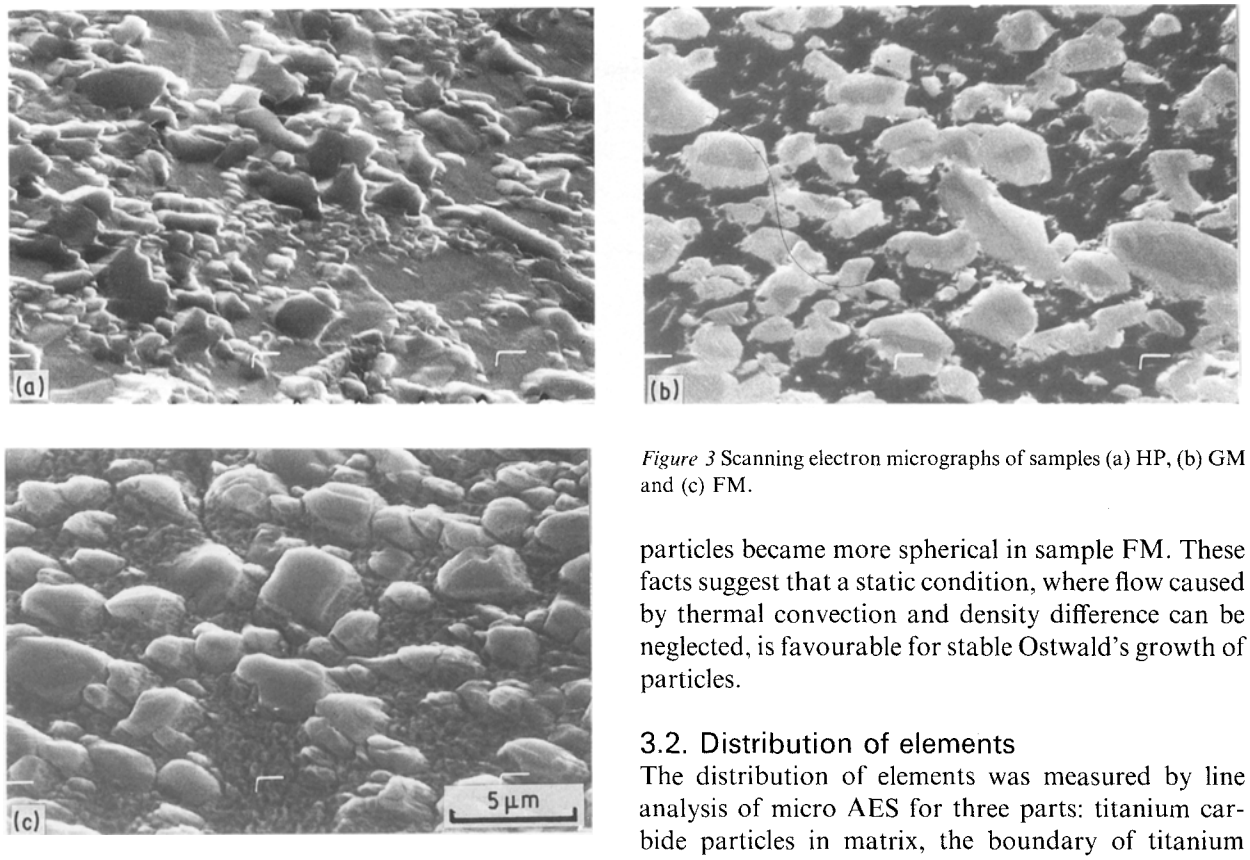


Figure 3 Scanning electron micrographs of samples (a) HP, (b) GM and (c) FM.

distribution of titanium carbide particles is much more uniform in sample FM than in sample GM. Figs 3a to c are SEM images of samples HP, GM and FM, respectively. In Fig. 3a, voids and irregular boundaries between titanium carbide and the matrix are observed. On the other hand, spherical titanium carbide particles are observed in Figs 3b and c. This difference is supposed to be due to the melting–pressing treatment, by which the titanium carbide particles are changed into spherical shapes with an increase in size.

Figs. 3b and c show scanning electron micrographs of the region where the distribution of titanium carbide particles was similar. It is recognized that there are irregularities in both size and shape of particles in the structure of sample GM. On the other hand, the particles became more spherical in sample FM. These facts suggest that a static condition, where flow caused by thermal convection and density difference can be neglected, is favourable for stable Ostwald's growth of particles.

3.2. Distribution of elements

The distribution of elements was measured by line analysis of micro AES for three parts: titanium carbide particles in matrix, the boundary of titanium carbide particles and their matrix, and the matrix.

Figs 4a to c show the spectrum of the above parts of samples HP, GM and FM, respectively. From Fig. 4a, the oxygen peak is recognized in titanium carbide particles of sample HP for the following reasons. (1) Oxygen and moisture were absorbed in the raw material powders. (2) They were taken up during the mixing process of the powders. (3) They were adsorbed on the surface because sample HP was a hot-pressed material and porous. A small amount of oxygen is observed for samples GM and FM in Figs. 4a and b which show the spectrum of titanium carbide particles and the surrounding area. The fact that oxygen was detected in both samples of GM and FM is due to use of sample HP as the starting sample.

Quantitative measurement of elemental distribution

TABLE I Measured values of the concentration and its standard deviation, σ, of titanium, carbon and nickel in the titanium carbide of samples HP, GM and FM

	Distance from the centre (μm)	HP		GM		FM	
		$\bar{x}$*	σ	$\bar{x}$	σ	$\bar{x}$	σ
Titanium	Centre	53.63	2.52	53.63	2.52	51.50	2.98
	0.6	49.12	1.40	44.88	5.00	49.52	2.60
	1.2	46.22	2.19	33.75	5.20	42.40	3.90
	1.8	44.92	2.01	20.75	5.18	34.45	8.40
	2.4	42.62	6.10	15.51	8.70	25.76	11.10
Carbon	Centre	46.37	2.54	45.77	2.67	47.70	3.68
	0.6	42.90	0.78	43.50	2.83	43.63	5.88
	1.2	40.93	1.04	35.88	4.19	41.86	8.20
	1.8	40.00	2.16	29.75	5.99	35.34	10.78
	2.4	40.31	4.22	29.54	10.60	29.50	11.45
Nickel	Centre			0.49	0.49	0.69	1.18
	0.6	7.98	0.54	8.81	2.79	5.29	4.32
	1.2	12.85	2.53	25.63	9.05	12.39	12.01
	1.8	15.10	3.83	43.59	12.86	24.99	17.07
	2.4	17.07	10.04	51.02	18.40	41.12	24.20

*$\bar{x}$ is the average value of concentration obtained from 90 points of micro AES measurement for sample HP and 150 points for samples GM and FM.

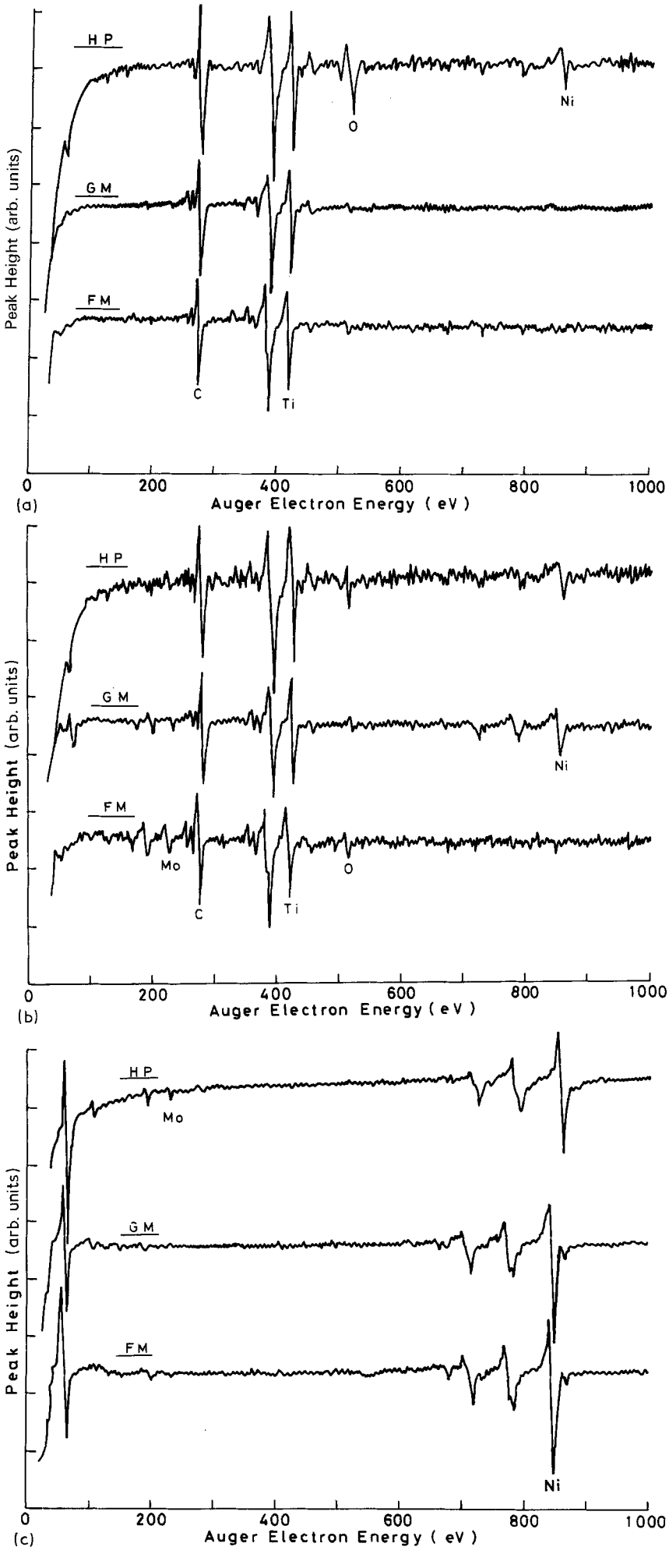


Figure 4 AES spectra of (a) titanium carbide, (b) the boundary of titanium carbide and (c) the matrix in the samples HP, GM and FM.

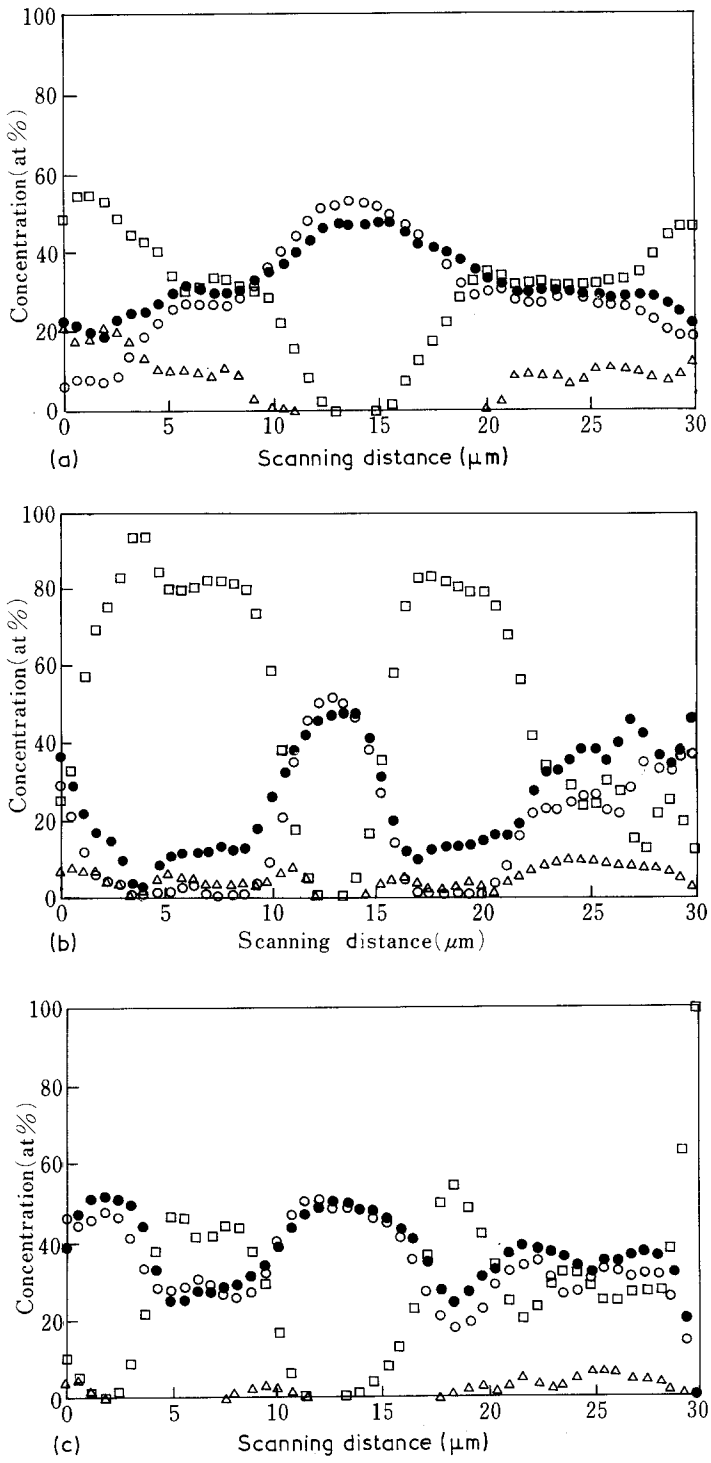
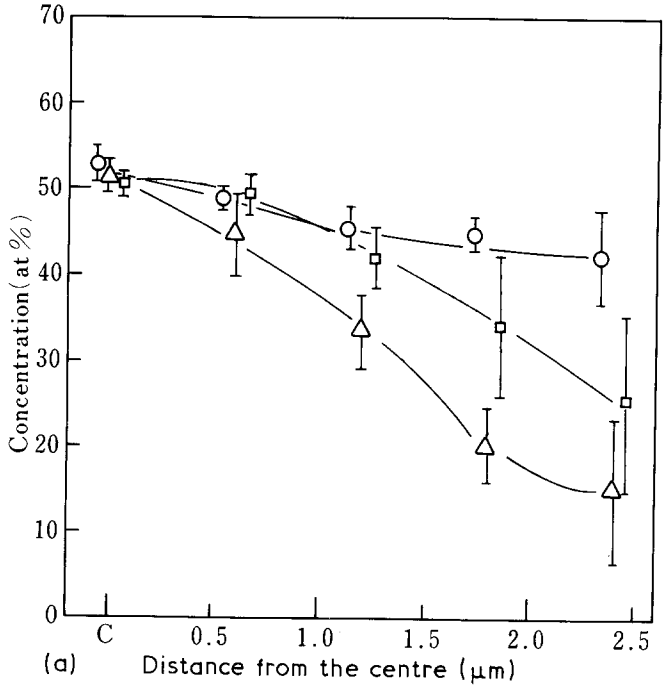


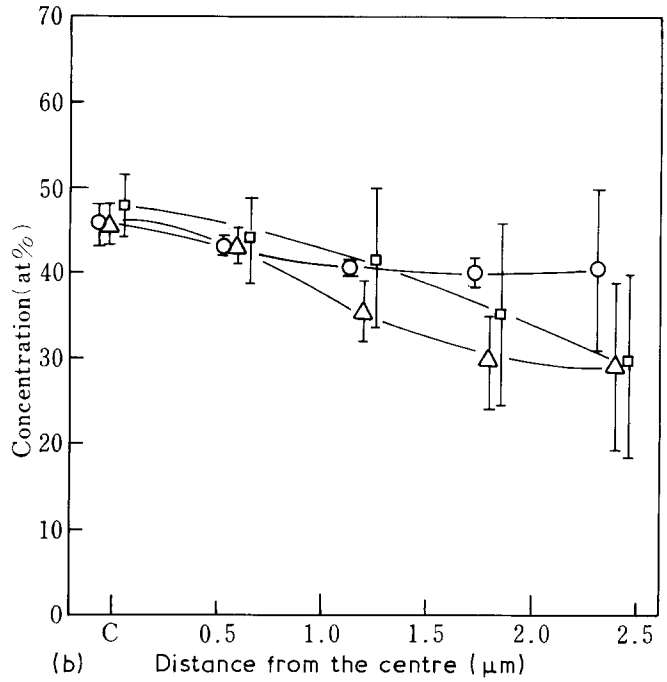
Figure 5 Elemental distribution along the line crossing a titanium carbide particle for samples (a) HP, (b) GM and (c) FM. (○) Ti, (●) C, (□) Ni, (△) Mo.

was carried out along a line crossing large titanium carbide particles of 3 μm or more in size. The results obtained are shown in Figs. 5a to c for samples HP, GM and FM, respectively. The plots in these figures indicate the average value of 90 line scans for sample HP and 150 scans for each of samples GM and FM. The diameter of the electron beam was about 0.3 μm as mentioned before.

The distribution curve of titanium and carbon elements corresponds to those of titanium carbide in sample HP (Fig. 5a) have a peak with a slope smoother than those of the other two samples. Such a situation seems to be due to the dispersion of extremely small particles of titanium carbide in the matrix of sample HP. Unlike the normal behaviour of the sample FM (Fig. 5c), the elemental distribution for sample GM (Fig. 5b) shows a more complex structure, implying the occurrence of violent flow of molten material.

The correlation of the distribution curves of titanium carbide with its constituent elements is easily understood in sample FM (Fig. 5c). The distribution curve of nickel in sample FM suggests a static diffusion reaction-controlled element migration in the sample which was kept in the molten state. The distributions of titanium, carbon and nickel elements in titanium carbide particles of each sample are shown in Fig. 6a to c with their deviations. The flat distribution curve of sample HP can be explained by the same reasons as described for Fig. 5a. It can be recognized from Fig. 6 that the migration of elements in titanium carbide particles in sample GM was faster than in sample FM under melting–pressing conditions, because of the higher nickel content, and lower





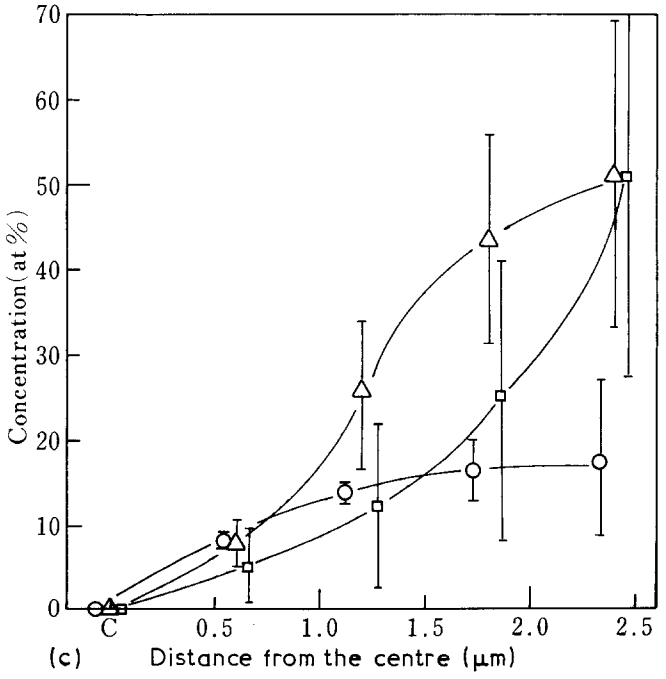


Figure 6 Distribution of (a) titanium, (b) carbon and (c) nickel in the titanium carbide particles dispersed in samples (○) HP, (△) GM and (□) FM.

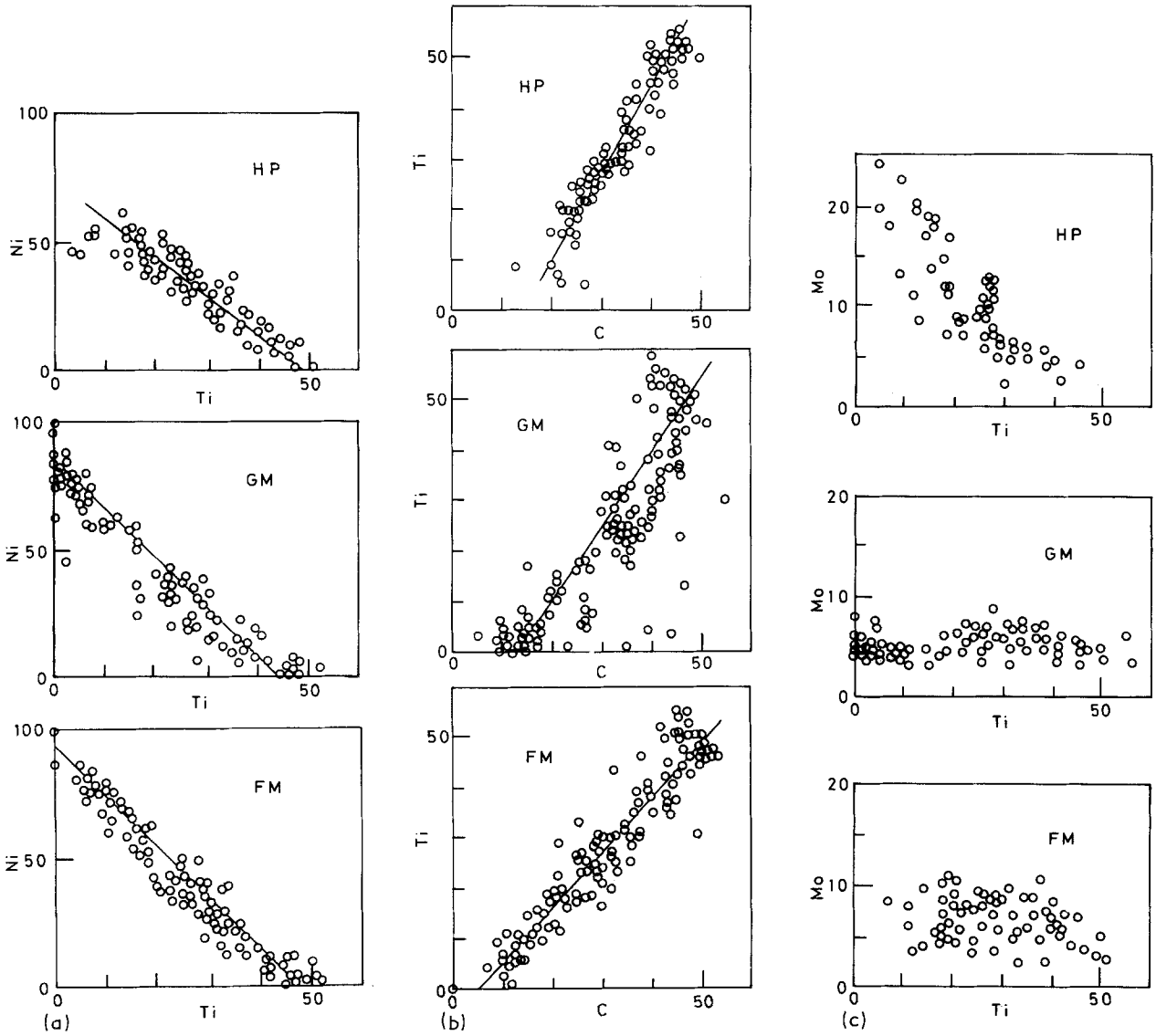


Figure 7 Correlation of elements (a) Ti–Ni, (b) C–Ti, and (c) Ti–Mo in samples HP, GM and FM.

contents of elemental titanium and carbon in the titanium carbide particle. Moreover, the distribution of elemental nickel in sample FM gave a suggestion of a diffusion reaction curve. Table I gives the data used for Fig. 6. The distance from the centre in Fig. 6 is the middle point of the scanned line of micro AES analysis for titanium carbide particle.

The concentrations of nickel, titanium, carbon and molybdenum were plotted in X–Y coordinates such as Ti–Ni, C–Ti and Ti–Mo to clarify the relationship of elements in the samples. The results are shown in Figs 7a to c for sample HP, GM and FM, respectively. A line showing the average measured points was drawn by the least squares method. Most of the plots of sample FM are on a linear line in (a). The site of 0% nickel and 50% titanium in coordinates corresponds to pure titanium carbide, and nickel is about 90% where titanium is 0%. These values are in good agreement with the chemical composition. A similar tendency may also be observed for sample GM. There are several 10% of scattering at each site near 90% nickel and 50% titanium for the Ti–Ni relation. The scattering is larger in the Ti–C relation of sample GM than those of the other two, as shown in Fig. 7b. The relation of Ti–Mo concentration is shown in Fig. 7c. Elemental molybdenum is distributed between zero and 50% titanium in sample HP and it is rich at the 0% titanium side. There is a similar tendency in sample FM; however, elemental molybdenum also diffuses at the 50% titanium side and concentrates in the region corresponding to the boundary between the titanium carbide particle and the matrix. On the other hand, elemental molybdenum is distributed everywhere between zero and 50% titanium in sample GM, i.e. an almost uniform distribution of elemental molybdenum can be seen over the whole range of this sample.

4. Conclusions

The microstructure and elemental distribution were analysed using microscopy and micro AES for three sample HP, titanium carbide particles turn into strengthened nickel molybdenum alloy. One was a hot-pressed sample of powder mixture (HP) and the other two samples were prepared through melting–pressing of sample HP on the ground (GM) or in space (FM).

1. Through the melting–pressing treatment of sample HP, titanium carbide particles turn into spherical shapes with a growth in size, and most of the

voids disappeared. The distribution of titanium carbide particles in sample FM was almost uniform, on the other hand, it was not so under the same situation in sample GM because of a density difference between titanium carbide and the molten matrix, and also the influence of thermal convection.

2. Three regions (titanium carbide particle, boundary of particle and matrix, and matrix) were analysed by micro AES using an electron beam of 0.3 μm diameter, and the distribution curves of the elements were obtained.

3. The elemental distribution curves showed that a mutual diffusion reaction at the interface between the particle and the matrix occurred during melting of the sample.

4. Molybdenum was rich at the periphery of the titanium carbide particle in sample FM, but the distribution was more even in sample GM.

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