

# Interdiffusion at the TiN/Ni interface in the cermet composed of TiN and Ni

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In order to investigate the interdiffusion at the TiN/Ni interface in the cermets based on Al<sub>2</sub>O<sub>3</sub> and nickel with a TiN interlayer, an independent experiment was designed and carried out. Pure TiN powder and pure nickel powder were selected as the starting materials. The cermet composed of TiN and nickel was manufactured by the powder metallurgy process including cold compacting and pressureless sintering at various temperatures in vacuum. During sintering, titanium atoms diffuse into nickel lattices forming homogeneous solid solution. This leads to the expansion of the lattice constant. The higher the sintering temperature, the more titanium atoms dissolve, and the more obvious is the expansion which occurs. Only a small amount of nickel diffuses into TiN and dissolves in it. The amount of the nickel is so small that the X-ray diffraction patterns show a stable crystal structure of TiN. ©1998 Kluwer Academic Publishers

## 1. Introduction

Various kinds of cermets are widely employed in industry owing to their favourable properties which are different from those of ceramics and metals. As one of their representatives, the cermet composed of WC and cobalt has been used as a wear-resistant material for several decades. However, this cermet is expensive because both tungsten and cobalt are not abundant materials. Furthermore, the high specific gravity of this material makes the parts too heavy to use in some cases, especially for aeronautical and astronautical applications. Hence, during the last 30y, many attempts have been made to find alternatives with low specific gravity and relatively comparable mechanical properties coupled with favourable cost [1–9]. Although much significant progress has been reported [1–9], the cermet composed of WC and cobalt has not so far been replaced in industry. Nevertheless, the attempts to seek alternatives have not stopped.

As reported elsewhere [10–12], in order to achieve this goal, Al<sub>2</sub>O<sub>3</sub> and nickel were selected as the starting materials owing to their favourable properties and easy availability. However, the problem associated with these two components is the poor wettability between solid Al<sub>2</sub>O<sub>3</sub> and liquid nickel [13], which hinders the manufacturing of this cermet by liquid-phase sintering. In order to improve the sinterability of this material, attention was focused on introducing interlayers which could adhere strongly to both the

ceramic phase and the metallic phase. It is reported that TiN was found to be the best suited to achieve this goal. A TiN interlayer was introduced by coating the Al<sub>2</sub>O<sub>3</sub> powder with a layer of TiN using the chemical vapour deposition (CVD) process. Then, Al<sub>2</sub>O<sub>3</sub> powder coated with TiN (denoted by Al<sub>2</sub>O<sub>3</sub>-TiN) was mixed with nickel powder. The mixture was hot pressed to manufacture the composite, denoted by Al<sub>2</sub>O<sub>3</sub>-TiN + Ni [10–12].

In order to investigate the mechanism by which the TiN interlayer remarkably enhances the sinterability and mechanical properties of the material, it is essential to study the two interfaces (TiN/Ni and Al<sub>2</sub>O<sub>3</sub>/TiN) involved in this system. This paper deals with the TiN/Ni interface.

## 2. Experimental procedure

Pure TiN powder (particle size 5–25 µm, purity 99.9 wt %) was mixed with pure nickel powder (particle size 1–3 µm, purity 99.9 wt %) at a volume ratio of 1 : 1 for 45 min using a powder mill. The powder was loaded in a plastic cylindrical vessel containing TiN balls, 10 mm diameter, for mixing. The mixed powder, 1 g for each sample, was cold compacted at 750 MPa for 60 s. Neither lubricant nor binder was used during compacting. The compacts were pressureless sintered at 1150, 1250 and 1350 °C respectively for 1 h in a vacuum of  $7 \times 10^{-3}$  Pa ( $5 \times 10^{-5}$  torr). After sintering for

the required duration, the samples were left in the furnace in vacuum to cool gradually to room temperature.

The sintered billets were ground lightly with 1000 mesh sand paper to remove the surface layer. Then they were washed in alcohol using an ultrasonic bath. After drying, X-ray diffraction was carried out on all these samples. The polished sections were observed by a scanning electron microscope (SEM) and the composition of the various phases at the sections was analysed qualitatively and quantitatively by an energy dispersive X-ray microanalysis system (EDX).

### 3. Results and discussion

Fig. 1 shows the X-ray diffraction (XRD) patterns of the samples sintered at the various temperatures. For comparison, the XRD pattern of the compact with the same composition is also included in the figure. This figure indicates that with increasing sintering temperature, every diffraction peak of nickel shifts to the left side of the figure (low angle), whereas the diffraction peaks of TiN do not shift. So, according to Bragg's law, the lattice constant,  $a$ , of nickel increases with increasing sintering temperature. Its value as a function of sintering temperature is presented in Fig. 2.

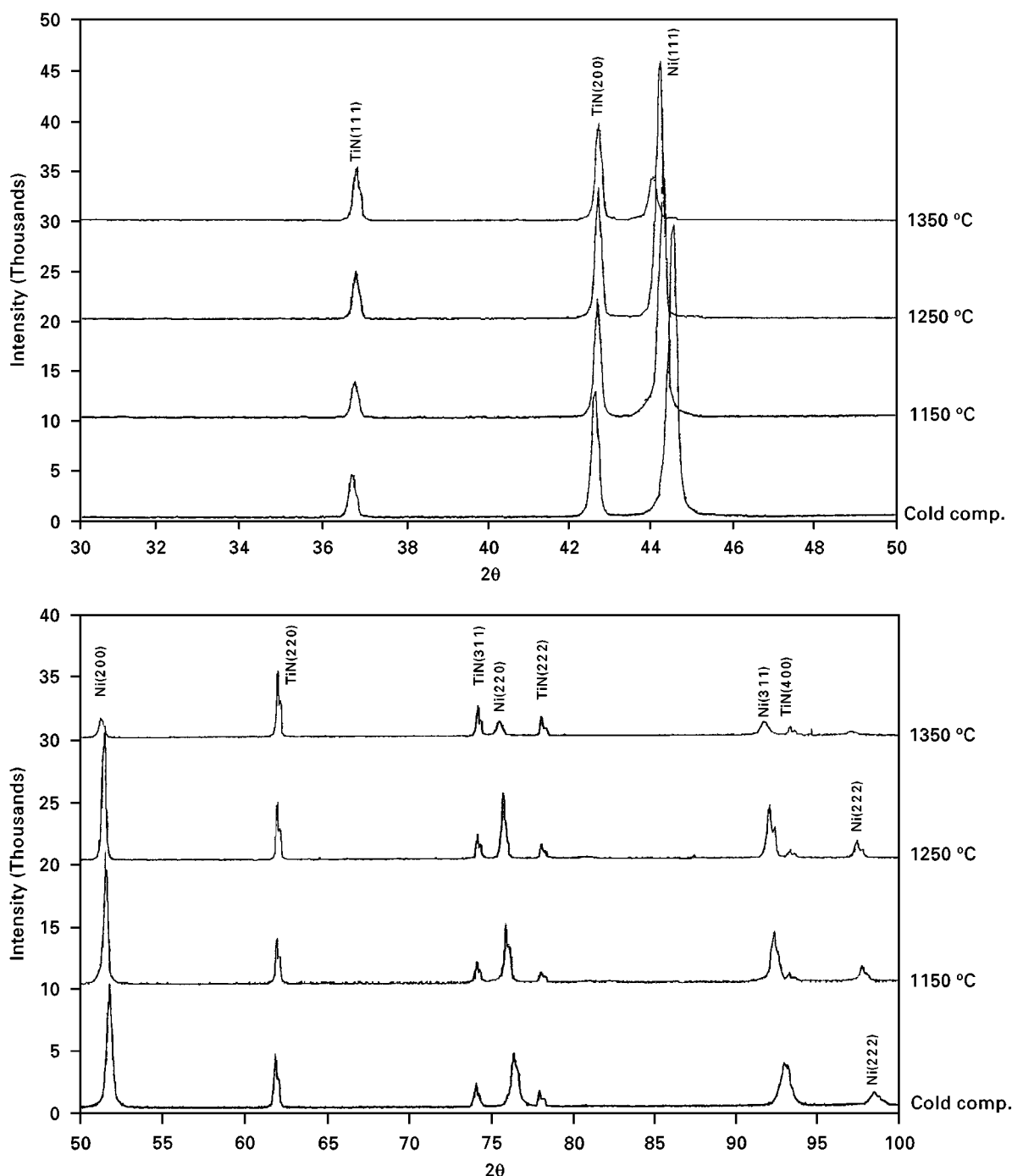


Figure 1 X-ray diffraction patterns of the cermets composed of 50 vol % TiN + 50 vol % Ni (a) before and (b) after pressureless sintering at various temperatures in vacuum for 1 h.

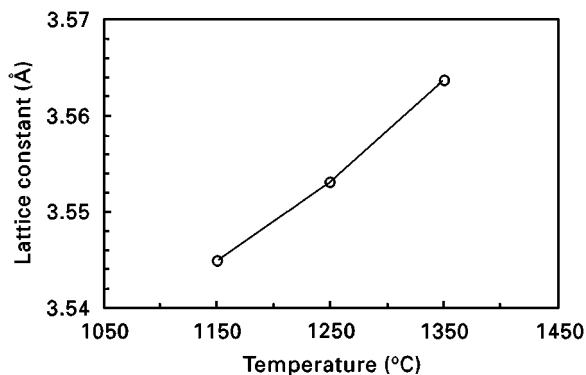


Figure 2 Lattice constant,  $a$ , of the nickel solid solution as a function of sintering temperature (for pure nickel  $a = 0.35238$  nm).

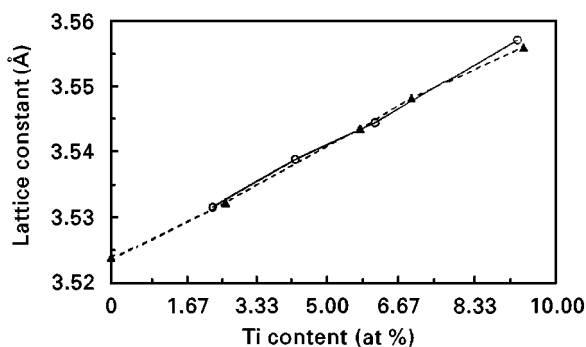


Figure 3 Lattice constant,  $a$ , of the nickel solid solution as a function of titanium content. (○) given by Poole and Hume-Rothery [X]; (▲) given by Taylor and Floyd [Y].

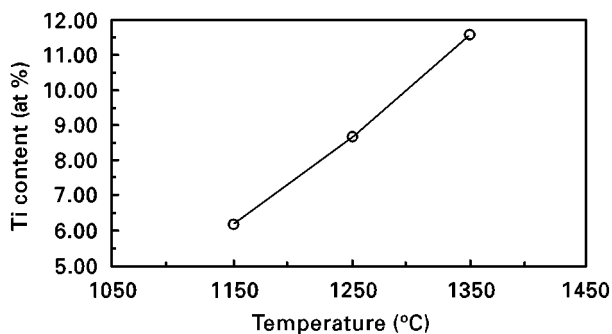


Figure 4 Titanium content of the nickel solid solution as a function of sintering temperature.

A conceivable explanation for the expansion is that dissociation of TiN takes place during sintering [14] resulting in the formation of titanium (or titanium ions), then the titanium atoms diffuse into nickel lattices leading to the expansion, as the atomic radius of titanium (0.2 nm) is larger than that of nickel (0.162 nm). The higher the sintering temperature, the more titanium atoms diffuse into nickel lattices and, consequently, the larger the lattice constant value will be. Fig. 1 also shows that there is no diffraction peak broadening, meaning that the nickel solid solution is homogeneous, which is not surprising for the high

temperatures and long duration of sintering applied and the small particle size of nickel.

The quantitative relationship between the lattice constant of nickel solid solution and its titanium content is given in Fig. 3 [15]. Using this figure, the titanium content of the nickel solid solutions can be obtained as illustrated in Fig. 4, which shows the titanium content as a function of sintering temperature acquired from the data presented in Fig. 2. From Fig. 4, one can see that the amount of titanium diffused into nickel lattices is significant.

Fig. 5 shows scanning electron micrographs of the cermet pressureless sintered at 1350 °C for 1 h. Two phases are visible in the micrographs. Various areas from each phase were analysed by EDX. A representative EDX spectrum for the metallic phase corresponding to the area indicated by the arrow in Fig. 5a is

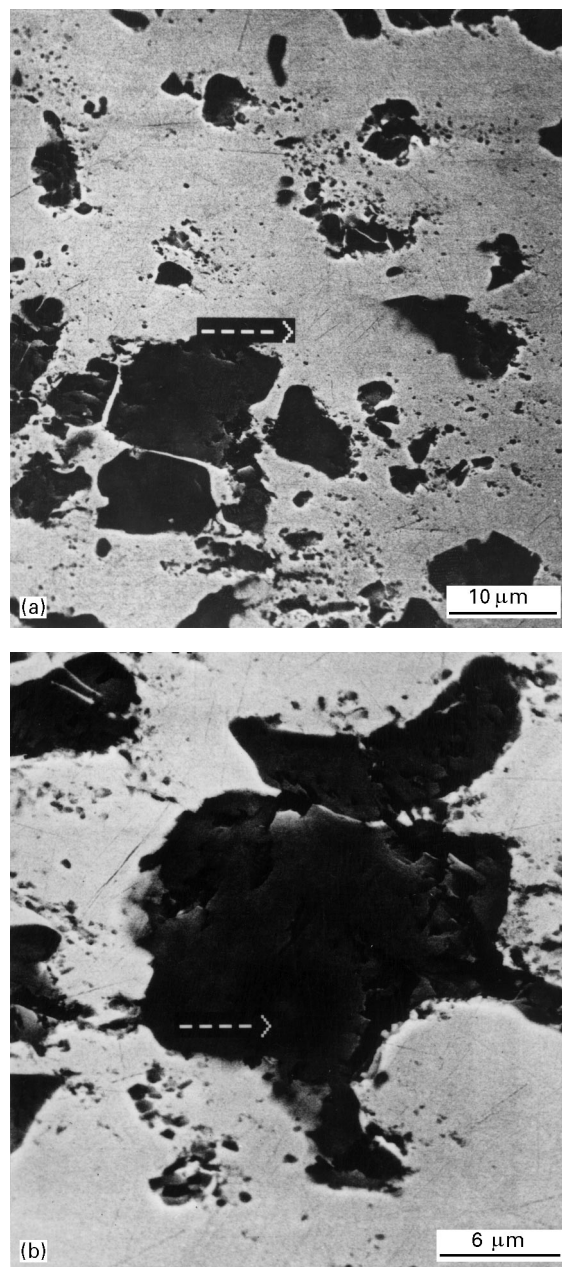


Figure 5 Scanning electron micrographs of the cermet composed of 50 vol % TiN + 50 vol % Ni pressureless sintered at 1350 °C in vacuum for 1 h. The metallic phase (a) and the ceramic phase (b) were analysed by EDX.

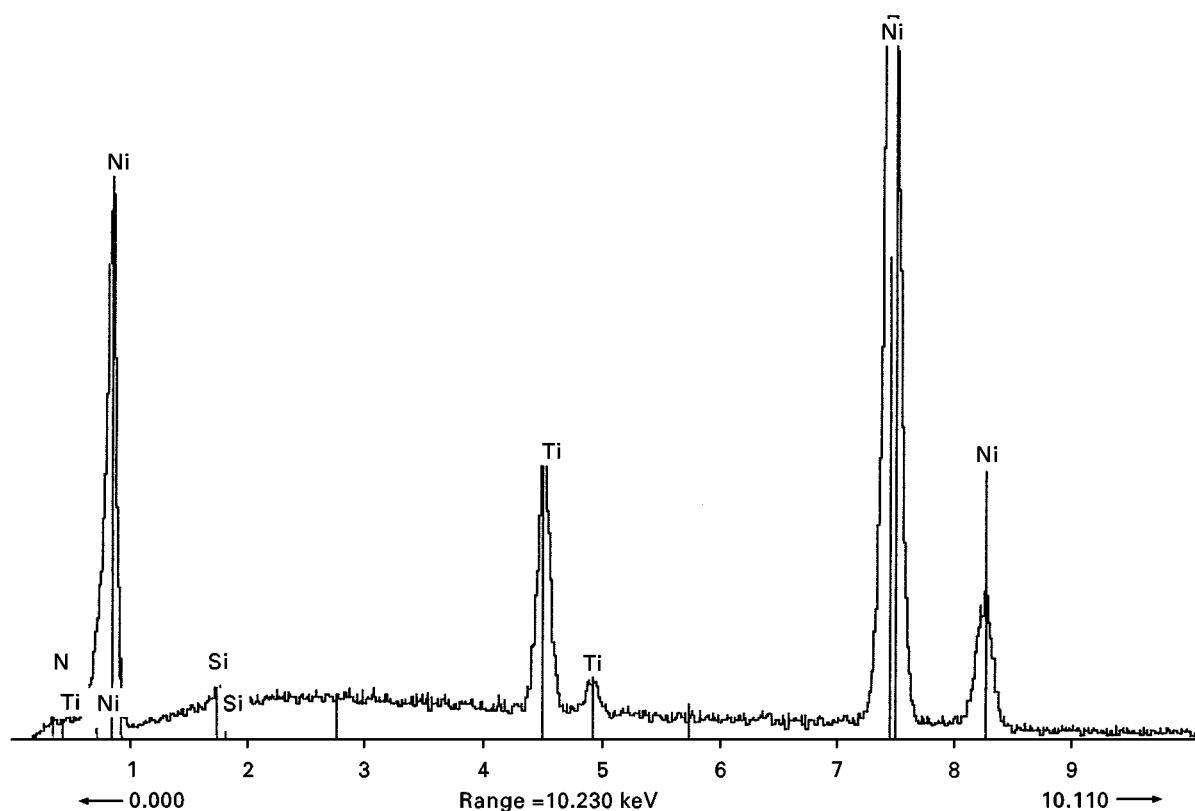


Figure 6 EDX spectrum analysed at the metallic phase as indicated by the arrow in Fig. 5a.

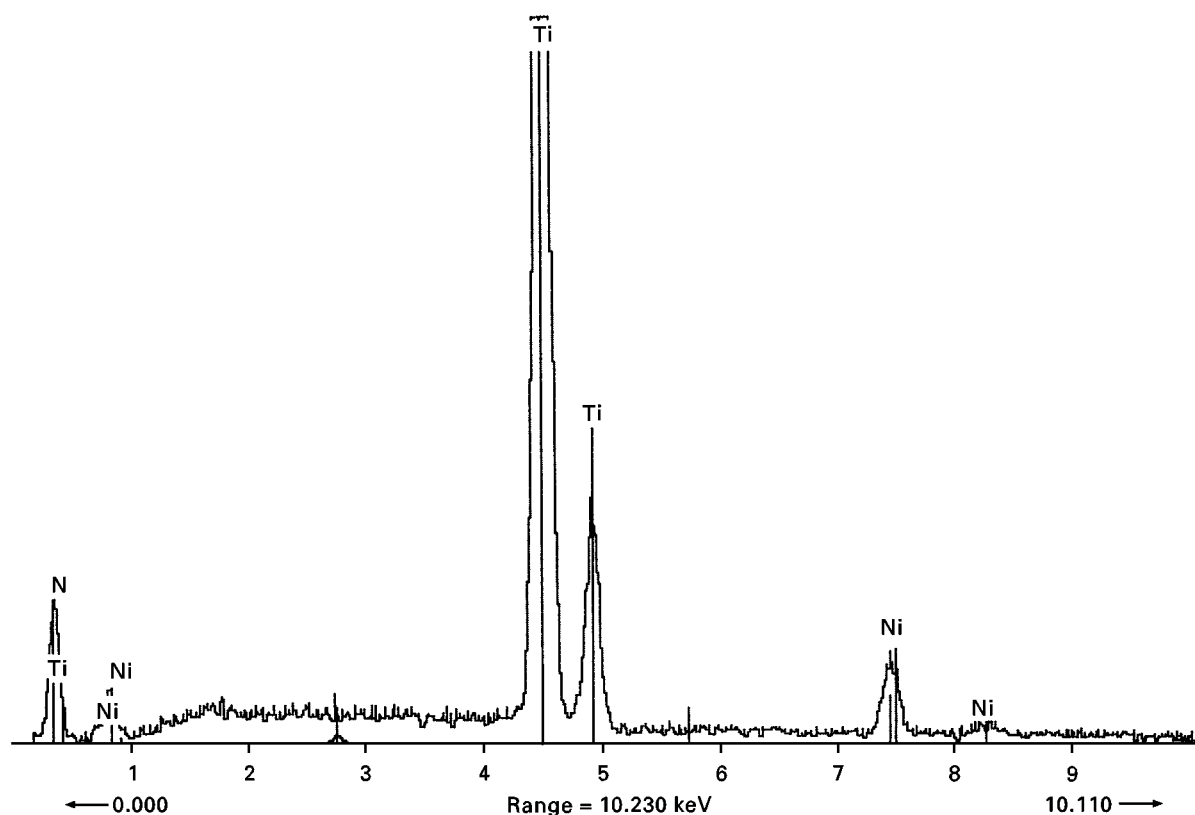


Figure 7 EDX spectrum analysed at the ceramic phase as indicated by the arrow in Fig. 5b.

presented in Fig. 6. This figure confirms that the metallic phase consists of nickel and titanium. The quantitative EDX analysis gives the titanium content as  $11.1 \pm 0.1$  at % (the uncertain range is the standard deviation). This value is very close to that given

in Fig. 4. A representative EDX spectrum for the ceramic phase corresponding to the area indicated by the arrow in Fig. 5b is presented in Fig. 7. This figure indicates that the ceramic phase consists of titanium, nitrogen and a small amount of nickel as

well. Quantitative EDX analysis shows that the nickel content is below 6 at %.

#### 4. Conclusion

Based on the experimental results, it can be concluded that during sintering of the compacts composed of 50 vol % TiN + 50 vol % Ni at 1150–1350 °C in vacuum for 1 h, titanium atoms diffuse into nickel lattices forming a homogeneous solid solution. This leads to the expansion of the lattice constant. The higher the sintering temperature, the more titanium atoms dissolve, and the more obvious is the expansion which occurs. Only a small amount of nickel diffuses into TiN and dissolves in it. The amount of the nickel is so small that the X-ray diffraction patterns show a stable crystal structure of TiN within the temperature range tested.

It is believed that this interdiffusion also takes place at the TiN/Ni interface involved in the  $\text{Al}_2\text{O}_3$ –TiN + Ni system. For the hot-pressed cermet  $\text{Al}_2\text{O}_3$ –TiN + Ni, this interdiffusion is beneficial to the adhesion at the TiN/Ni interface. Therefore, it may be an explanation of the mechanism by which the TiN interlayer remarkably enhances the sinterability and mechanical properties of the  $\text{Al}_2\text{O}_3$ –TiN + Ni system.

#### Acknowledgements

The authors gratefully acknowledge the financial support of the project by the former Foundation for Advanced Metals Science, The Netherlands, The University of Twente, The Netherlands, and the National Natural Science Foundation of China.

The authors thank Mr. M. A. Smithers for his help and assistance with the SEM work.

#### References

1. C. O. McHUGH, T. J. WHALEN and M. HUMENIK, *J. Am. Ceram. Soc.* **49** (1966) 486.
2. G. E. GAZZA, J. R. BARFIELD and D. L. PREAS, *Am. Ceram. Soc. Bull.* **48** (1969) 606.
3. D. T. RANKIN, J. J. STIGLICH, D. R. PETRAK and R. RUH, *J. Am. Ceram. Soc.* **54** (1971) 277.
4. E. D. WHITNEY, *Powder Metall. Int.* **10** (1) (1978) 16.
5. Y. NAERHEIM, *ibid.* **18** (3) (1986) 158.
6. R. J. BROOK, in "High Tech Ceramics", edited by P. Vincenzini (Elsevier Science, Amsterdam, 1987) p. 757.
7. R. TRABELSI, D. TREHEUX, D. GOEURLOT-LAUNAY, P. GOEURLOT, F. THEVENOT, G. ORANGE and G. FANTOZZI, *ibid.*, p. 2683.
8. M. LEE and M. P. BOROM, *Adv. Ceram. Mater.* **3** (1) (1988) 38.
9. P. GOEURLOT, D. GOEURLOT-LAUNAY and F. THEVENOT, *J. Mater. Sci.* **25** (1990) 654.
10. SHUIE LI, in "Investigation of Powder Metallurgical Route for Manufacturing  $\text{Al}_2\text{O}_3$ /Ni Cermets with Interlayers" (Quick Service drukkerijen Nederland B.V., Enschede, The Netherlands, 1992) p. 3.
11. SHUIE LI, P. B. KHOSROVABADI and B. H. KOLSTER, *Int. J. Refract. Metals Hard Mater.* **11** (3) (1992) 181.
12. *Idem*, *ibid.* **11** (3) (1992) 193.
13. U. M. FEIDORLQINK, in "Principles of Powder Metallurgy" (The Publishing House for Metallurgical Industry, Beijing, 1974) p. 329 (Chinese translation from the Russian).
14. E. D. MNATSAKANYAN, YU. N. VIL'K, YU. N. PAPUTSKII, L. V. KOZLOVSKII and S. S. ORDAN'YAN, *Sov. Powder Metall. Met. Ceram* **24** (1985) 812 (English Translation).
15. W. B. PEARSON, in "A Handbook of Lattice Spacings and Structures of Metals and Alloys" (Pergamon Press, Oxford, London, Edinburgh, New York, Paris, Frankfurt, Reprinted with corrections 1964) p. 779, 791.

Received 14 October 1997  
and accepted 15 May 1998