Effect of a Titanium Nitride Interlayer on the Densification, Properties and Microstructure of Cermets Based on Alumina and Nickel — Part 2: Microstructure

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Abstract: SEM microstructural analyses in conjunction with EDX and TEM microstructural analyses have been conducted with cermets based on nickel and alumina, the latter as such and with a chemical-vapour-deposited titanium nitride layer. It has been proved that there is excellent bonding at both the Al₂O₃/TiN and the TiN/Ni interface, whereas Al₂O₃ and Ni do not adhere to each other. This is the reason for the observation that the mechanical properties as well as the densification of cermets consisting of Al₂O₃ and Ni are enhanced by applying a TiN interlayer between the ceramic phase and the metallic phase.

1 INTRODUCTION

In a previous paper, it was concluded that applying a TiN interlayer between Al₂O₃ and Ni contributes to the properties of cermets based on these two constituents. Both the relative density and the mechanical properties of the cermets improve due to the presence of this interlayer. In order to explain this effect of the TiN interlayer, the microstructure of the cermets have been studied in detail.

2 EXPERIMENTAL PROCEDURES

As mentioned in Ref. 1, the compositions of the studied cermets are 48.9 vol. % Al₂O₃-TiN + 51.1 vol. % Ni and 48.9 vol. % Al₂O₃ + 51.1 vol. % Ni. (In this paper, Al₂O₃-TiN represents Al₂O₃ powder coated with TiN. This powder was obtained from Xycarb, one of the partners of the Brite-Euram project ‘Sintering of coated ceramic powders for wear and fatigue resistant components’, Dutch patent No. 9000346.) The cermets were densified by hot-pressing at 1150°C or 1200°C, at 41 MPa for 60 min in a vacuum of 7 x 10⁻³-7 x 10⁻⁴ Pa (5 x 10⁻⁵-5 x 10⁻⁶ torr), or by pressureless sintering at 1150°C or 1350°C for 60 min in a vacuum of 7 x 10⁻²-7 x 10⁻⁴ Pa (5 x 10⁻⁴-5 x 10⁻⁶ torr). After measuring the density, compressive tests were conducted to determine the mechanical properties of the cermets. The fracture surface was analyzed by SEM and EDX. Before the compressive tests, the hot-pressed billets were ground and polished. Then, the microstructures and compositions of the samples were examined by SEM and EDX. The microstructures of the cermets were also characterized by TEM before the compressive tests.

3 RESULTS AND DISCUSSION

3.1 Observations at polished sections by SEM and EDX

Figure 1 shows the scanning electron micrograph of the polished cross-section of the cermet, with-
out the TiN interlayer, hot-pressed at 1150°C and 41 MPa for 1 h. The dark area represents Al₂O₃ and the bright area represents Ni. It can be seen in this photograph that there are many pores and gaps at the interface between the Al₂O₃ phase and the Ni phase. Figure 2 shows the scanning electron micrograph of the polished cross-section of the cermet with the TiN interlayer, hot-pressed at 1150°C and 41 MPa for 1 h. No pores and gaps at the interface between the ceramic phase and the metallic phase are seen. The interface area indicated by the arrow was analyzed by EDX. Figure 3 shows the spectrum corresponding to Fig. 2. This spectrum indicates that there exists Ti (from TiN) at the interface. Figure 4 shows two other scanning electron micrographs of this sample, revealing the presence of pores at the interface areas indicated by the arrows. Figure 5 presents

**Fig. 1.** Scanning electron micrograph of polished section of the cermet composed of 48.9 vol. % Al₂O₃ + 51.1 vol. % Ni (without interlayer) hot-pressed at 1150°C and 41 MPa for 1 h. Many pores and gaps exist at the interface.

**Fig. 2.** Scanning electron micrograph of polished section of the cermet composed of 48.9 vol. % Al₂O₃-TiN + 51.1 vol. % Ni (with interlayer) hot-pressed at 1150°C and 41 MPa for 1 h. No pores and gaps exist at the interface.

**Fig. 3.** EDX spectrum analyzed at the area indicated by the arrow in Fig. 2.
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Fig. 4. Scanning electron micrographs of polished section of the cermet composed of 48.9 vol. % Al₂O₃-TiN + 51.1 vol. % Ni hot pressed at 1150°C and 41 MPa for 1 h. There are pores at the interface.

Fig. 5. EDX spectrum analyzed at the area indicated by the arrow in Fig. 4(a).

the EDX spectrum analyzed at the area indicated by the arrow in Fig. 4(a). The spectrum analyzed at the area indicated by the arrow in Fig. 4(b) is similar to Fig. 5 and is omitted in this paper. These spectra indicate that no Ti is present at these areas. This is due to the poor coating in these regions. Taking into account that there are uncoated surface regions and holes in the coating layer of the powder (see Figs 4–6 in Ref. 1), these results are reasonable.

Many areas at the interface of the hot-pressed cermets have been analyzed by SEM and EDX. Almost no pores and gaps were found in regions of the interface containing the TiN interlayer.
Pores and gaps were only seen at those areas where no TiN exists. These results indicate that the TiN interlayer can significantly decrease or eliminate the gaps and pores at the interface between the ceramic phase and the metallic phase. Consequently, the relative density and the mechanical properties of the cermet are improved by applying the TiN interlayer.

Fig. 6. Scanning electron micrographs of the fracture surface of hot-pressed cermet composed of 48.9 vol. % Al₂O₃ + 51.1 vol. % Ni. (a) 1200°C, 41 MPa, 60 min (b) 1150°C, 41 MPa, 60 min.

Fig. 7. Scanning electron micrographs of the fracture surface of pressureless sintered cermet composed of 48.9 vol. % Al₂O₃ + 51.1 vol. % Ni. (a) 1150°C, 60 min; (b) 1350°C, 60 min.
3.2 Observations by SEM and EDX at fracture surfaces

Figure 6 shows two scanning electron micrographs of the fracture surface of the $\text{Al}_2\text{O}_3 + \text{Ni}$ cermet hot-pressed at $1200^\circ$C and $1150^\circ$C, respectively (after the compressive test). These photographs reveal that $\text{Al}_2\text{O}_3$ and Ni do not adhere well to each other. Figure 7 shows two scanning electron micrographs of the fracture surface of the $\text{Al}_2\text{O}_3 + \text{Ni}$ cermet, pressureless sintered at $1150^\circ$C and $1350^\circ$C, respectively. These two pictures reveal poor densification and also bad adhesion between the $\text{Al}_2\text{O}_3$ phase and the Ni phase.

The scanning electron micrograph of the fracture surface of the pressureless sintered $\text{Al}_2\text{O}_3$-TiN + Ni cermet is shown in Fig. 8. It is clear that a lot of particles stick on the $\text{Al}_2\text{O}_3$-TiN surface. The areas indicated by the arrows were analyzed by EDX. Almost the same spectra were obtained from the three different areas, one of which is shown in Fig. 9. This spectrum shows that the analyzed area contains Al, Ni and Ti, indicating that the particles on the $\text{Al}_2\text{O}_3$-TiN surface are Ni particles. Further evidence can be found in Fig. 10, showing another scanning electron micrograph and the corresponding X-ray images of this sample. This figure also shows that most of the $\text{Al}_2\text{O}_3$ surface is coated with TiN. It can also be seen that many small Ni particles stick to the TiN layer, but none to the uncoated $\text{Al}_2\text{O}_3$ surface, as is revealed from the upper left corner in Fig. 10. (It should be recalled that about 3% of

![Fig. 8. Scanning electron micrograph of the fracture surface of pressureless sintered (at 1350°C for 60 min) cermet composed of 48.9 vol. % $\text{Al}_2\text{O}_3$-TiN + 51.1 vol. % Ni.](image)

![Fig. 9. EDX spectrum analyzed at the areas indicated by the arrows in Fig. 8.](image)
the $\text{Al}_2\text{O}_3$ surface is not coated). It cannot be ruled out, however, that the uncoated area in Fig. 10 is the fracture surface of a fractured $\text{Al}_2\text{O}_3$ particle. Even so, Fig. 10 proves that Ni particles firmly stick to the TiN coating layer. Figure 11 shows another scanning electron micrograph of this sample at high magnification. The photograph reveals excellent adhesion between TiN and Ni as well as between $\text{Al}_2\text{O}_3$ and TiN.

Figure 12 is a scanning electron micrograph of the fracture surface of a hot-pressed $\text{Al}_2\text{O}_3$-TiN+Ni cermet. In this picture, it can be seen that a coated $\text{Al}_2\text{O}_3$ particle (at the bottom left corner of the picture) adheres well to Ni, unlike the uncoated $\text{Al}_2\text{O}_3$ particle in the middle of the picture.

### 3.3 Observations by TEM

The TEM picture in Fig. 13 also reveals a gap at the interface for the pressureless sintered $\text{Al}_2\text{O}_3$+Ni cermet. Hot-pressing does not result in a better adhesion for this cermet as shown in Fig. 14: a gap is again visible at the $\text{Al}_2\text{O}_3$/Ni
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Fig. 13. TEM photograph of cermet composed of 48.9 vol. % Al₂O₃ + 51.1 vol. % Ni pressureless sintered at 1350°C for 60 min.

Fig. 14. TEM photograph of cermet composed of 48.9 vol. % Al₂O₃ + 51.1 vol. % Ni hot-pressed at 1200°C and 41 MPa for 60 min.

Fig. 15. TEM photographs of cermet composed of 48.9 vol. % Al₂O₃-TiN + 51.1 vol. % Ni hot-pressed at 1200°C and 41 MPa for 60 min.

Fig. 16. TEM photographs of cermet composed of 48.9 vol. % Al₂O₃-TiN + 51.1 vol. % Ni hot-pressed at 1200°C and 41 MPa for 60 min.

interface. So, also, the TEM investigations show that Al₂O₃ and Ni do not adhere to each other during either pressureless sintering or hot-pressing.

TEM photographs of the hot-pressed Al₂O₃-TiN + Ni cermet are shown in Fig. 15. Photographs (a) and (b) were taken from the same area of the sample but with a different contrast. From these two photographs it is clear that the bright areas at the interface are not pores but small grains with different orientation. In addition, these two pictures show that the thickness of the TiN interlayer is about 0.3 μm. The pictures also show excellent bonding at both the Al₂O₃/TiN and the TiN/Ni interface. Also, at high magnification a perfect adhesion between Al₂O₃ and TiN as well as between TiN and Ni can be observed (Fig. 16).

From the microstructure of the cermets presented in this paper, it is conceivable that the pores and gaps at the Al₂O₃/Ni interface cause a slight decrease in relative density, but result in a significant degradation in the bonding at the inter-
face. This is a reason for the observation that a slight increment in the relative density of the cermet by applying the TiN interlayer is accompanied by a pronounced increment in the mechanical properties as described in the previous paper.¹

4 CONCLUSIONS

— A TiN interlayer can significantly decrease or eliminate the gaps and pores at the metallic/ceramic interface. This causes a slight increase in relative density and a significant improvement in bonding at the interface.

— Scanning and transmission electron microscopy reveal excellent bonding at both the $\text{Al}_2\text{O}_3/$TiN and the TiN/ Ni interface of pressureless sintered and the hot-pressed $\text{Al}_2\text{O}_3$-$\text{TiN}$ + Ni cerments; on the other hand, $\text{Al}_2\text{O}_3$ and Ni do not adhere well to each other, as could be concluded from the microstructure of the pressureless sintered and the hot-pressed $\text{Al}_2\text{O}_3$ + Ni cerments. The observed difference in mechanical properties as well as in densification between the two cerments is no doubt due to these different bonding characteristics. In other words, favourable properties for cerments based on $\text{Al}_2\text{O}_3$ and Ni can be obtained by applying a coating on $\text{Al}_2\text{O}_3$ particles, which adheres both with $\text{Al}_2\text{O}_3$ and with Ni.

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REFERENCE