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Oak Ridge National Laboratory
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ABSTRACT

Aluminide-bonded ceramics (ABC's), because of their high-temperature properties, are attractive for use in diesel engine applications and for other applications where high-temperature wear resistance and strength are requirements. Issues related to the application of these "new" cermets include: cost effective processing, alloying systems and additive element effects, near-net-shape fabrication, and enhanced manufacturing techniques. Typically for cermets, binder contents of 10 to 30 vol. % are used, however binders contents of 30 to 40 vol% may be necessary to match the thermal expansion coefficient of steel. Continuous sintering has been found to be a cost effective and often beneficial technique for producing engineering ceramics with enhance properties, but has not been investigated for ABC's.

In this work nickel-aluminide and iron-aluminide bonded TiC, with 30 vol% binder containing alloy modifying elements of up to 20 atom %, were investigated. The modifying elements being investigated include: Fe, Cr, Si, Ti, Mo, W, Co, and Zr.

As part of this study, density, microhardness, indentation fracture toughness and microstructure obtained by pressure sintering under 10 atm of Ar and by continuous sintering in flowing Ar were examined. These results will be presented and discussed.

INTRODUCTION AND LITERATURE SEARCH

Previous investigations have shown that silicon nitride (Si_3N_4) is a very usable material for various engine parts.[1] Si_3N_4 is used for turbochargers, cam followers and valve seats, valves, bearings and other applications. Recently, aluminide-bonded cermets have become of interest for some of these parts. These materials have advantages of very high wear resistance and high hardness. Some of the currently used materials for diesel engine applications are limited by their relatively low strength, oxidation and corrosion resistance at elevated temperatures, and might benefit from replacement of current materials with cermets.

In order to make some cermets competitive as structural materials with the currently used metals and super alloys, the total material and production costs must be similar or lower than for the metal parts.

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Use of lower cost binders, machining cost and sintering costs are areas where cost reduction is possible. In studies performed to reduce the processing cost of Si_3N_4 by continuous sintering in N_2, it was demonstrated that near full density materials could be produced by continuous sintering at about a 50% reduction in the cost of sintering.[2] In addition, the properties of continuously sintered Si_3N_4 were often equal to or better than those for the same Si_3N_4 produced by batch sintering.[3] Because of these successes, it was thought to be feasible that continuous sintering could be used to produce high-density cermets with satisfactory properties.

Another area where cost reduction is possible is in the binder material. The most common binder for current cermets is Co, which is costly. Co is also a strategic material, since there is no domestic source for Co containing ore. Further, for applications in diesel engines, a thermal expansion coefficient similar to that of cast iron is desired, and traditional Co bonded cermets have lower thermal expansion than steel.[7] There has also been an interest in introducing cermets into radioactive environments, but since Co decays into reaction products with long half-life, these materials are not suitable for these applications. Finding binders with lower cost is therefore very important to reduction of the total cost of these materials. Because many cermets are bonded with Co raises some concern regarding the material properties as well. These materials have:

- poor corrosion and oxidation resistance in certain solutions; [4-8]
- strength that decreases drastically with temperature; [9] and
- high brittleness.

Alternative binders have been investigated with promising results. Binders with the most promising properties are FeAl and Ni_3Al. By introducing Ni_3Al, better high-temperature properties were obtained, since Ni_3Al has the unusual behavior of increasing strength up to about 800°C.[6] It is well known that Ni_3Al is very brittle in the polycrystalline form, due to weaknesses in the grain boundaries, but by doping the Ni_3Al with boron the ductility of Ni_3Al is increased significantly.[11] Very small amounts of B (0.01 - 0.4 at. %) and aluminum contents of less than 25 vol.% proved to increase the ductility of the aluminides significantly. By introducing boron-doped Ni_3Al into TiC cermets the ductility was increased.[11,12] Fracture toughness and hardness have been found to be equal or better than Co-based cermets in several investigations.[13,14] FeAl bonded WC and TiC have been reported to have better oxidation resistance in air than Co bonded WC and better wear resistance.[7] The hardness for FeAl bonded cermets has been shown to increase initially, as the carbide volume fraction was increased, and then decrease with further increase in carbide content.[5,7]

It is well known that the most prominent mechanism for sintering of carbides with metal or intermetallic binder is liquid phase sintering. For this sintering method the wetting of the carbide powder by the metal or intermetallic is of great importance for densification and final properties. Carbides, such as WC and TiC are known to be wetted well by aluminides, and a number of different sintering methods can be used. It has been shown that by using different sintering techniques it is possible to obtain different properties for similar material compositions.[7,12,14,15]

Research over the last decade has focused on improvements in ductility, high-temperature properties, hardness and flexural strength of cermets. These goals have been partially accomplished by the introduction of aluminide-based bonding materials. The sintering techniques that have been used are hot pressing, vacuum sintering, pressureless sintering and melt infiltration. Until this time, continuous sintering in flowing Ar has not been reported as a potential sintering technique.

Hot pressing of WC and TiC with Ni_3Al has been shown to give high densities and good mechanical properties. Several investigations have shown that the binder content highly influences the sintering behavior.[4,5,12,13,15-17] Low densities were obtained for low binder contents (<5 vol.%) at any sintering temperature for WC- Ni_3Al. Binder contents of >10 vol.% and temperatures as low as 1300°C, produced close to theoretical densities.[13] At higher sintering temperatures, but below 1450°C, carbon precipitates could be detected by SEM in the WC cermets.[4,12] Ni_3Al-TiC cermets (using prealloyed Ni_3Al) did not show any precipitation.

Early studies in binder composition showed that sintering behavior and properties, predominantly flexural strength, were highly affected by small changes in composition. Several investigations used three different commercial Ni_3Al compositions[12,13], as the binder for TiC. The Ni_3Al compositions are given in Table I. Flexural strength increased by about 200% when using IC-218, which contains Cr and Zr, instead of IC-15 which is a baseline boron doped Ni_3Al.[12] Hardness and fracture toughness were not affected nearly as much, but fracture toughness was reduced with an increase in alloying elements (from IC-15 to IC-218).

Table I. Ni_3Al alloy compositions for hot-pressed powders[12]

Alloy	Al	B	Zr	Cr	Ni
IC-15	12.7	0.05	--	--	Bal
IC-50	11.3	0.02	0.6	--	Bal
IC-218	8.5	0.02	0.8	7.8	Bal

Pressureless sintering of WC and TiC proved to give high densities for higher temperatures, ~ 1500°C and higher binder fraction, ~ 20 vol.% than hot-pressing.[12] The temperatures required for acceptable densification of TiC were less than those for WC based cermets. By introducing Fe into the binder phase the flexural strength was increased, but W and Ti additions were observed to decreased the flexural strength. Overall, the flexural strength was lower than for hot pressed materials. Ti, on the other hand enhanced the densification. Hardness for pressureless sintered TiC was proven to be better than, or equal to hot-pressed TiC. Hardness was increased by all three binder compositions.

The same results were found for vacuum sintered TiC.[16] For vacuum sintered TiC the strength was found to be similar to hot pressed TiC.

Melt infiltration of Ni_3Al and FeAl in WC and TiC has produced very high densities, >97% for high ceramic contents and relatively low temperatures, similar to those used in hot pressing.[5,12,15] Densities over 98% of theoretical are achieved for Ni_3Al contents as low as 8 vol.% using this method.[15] The final material did not show any incorporation of Ni or Al into the TiC. The increase in densification could be due to several reasons.[12] First, the Ni_3Al powder was not milled, which could reduce the oxide formed. Secondly, the Ni_3Al powder used in this investigation was coarser than the powder used for hot pressing and pressureless sintering; this might have reduced the oxidation even further. The microstructure was more uniform, and it showed a continuous binder phase.[5]

Results from these investigations suggest that sintering behavior and mechanical properties can be altered by: ceramic volume fraction, binder composition, sintering technique; and sintering parameters. Further research with regard to these factors needs to be performed in order to find the most suitable composition and sintering technique to achieve the goals of: (1) cost effective material with high-temperature properties better than WC-Co and (2) thermal expansion coefficient match with cast iron.

The focus of this work was to investigate the effect of sintering methods on the properties and microstructure of TiC cermets, using Ni_3Al alloys for the binder phase.

PROCEDURE

The physical characteristics of the powders used in fabrication of the composites are given in Table II. Note the large size of the pre-alloyed Ni_3Al powder produced by inert gas atomization compared to the

other powders. Samples were fabricated by two different methods: (1) sintering with pre-alloyed gas-atomized Ni_3Al, or (2) reaction sintering with fine elemental powders to form Ni_3Al alloys in-situ. Table III gives the composition of the intermetallic systems investigated. Sintering was then accomplished by either batch sintering or continuous sintering.

Table II. Powder identification and information.

Powder Type	Supplier	Grade	Ave. Particle Diameter (μm)
TiC	Kennametal, Latrobe, PA	TICA3	1.3
Ni	Novamet Wyckoff, NJ	Type 123	5
NiAl	X-Form Cohoes, NY	--	10.9
B	Cerac Milwaukee, WI	–	0.3
Ni_3Al	Homogeneous Metals Clayville, NY	IC-50	<44
Fe, Co, Si, Cr, Mo	Alpha Ward Hill, MA	–	<5
W	Consolidated Astonautics Saddlebrook, NJ	–	1
Ti	Atlantic Equip. Eng. Bergenfield, NJ	–	70

The billets using pre-alloyed intermetallic powders were fabricated by ball milling fine TiC powder, with pre-alloyed Ni_3Al powder. The milling was done in isopropanol for 16 h, using WC-Co milling media with 1 wt.% PVP* added as a binder. The reaction sintered materials were fabricated by ball milling appropriate amounts of TiC, Ni, NiAl, and B together to form Ni_3Al in-situ as a reaction product. In addition, some composites were fabricated with alloying additives, such as Fe, Si, W, Ti, Co, Cr, and Zr. All of the compositions used a 0.1 wt. % boron addition, which was added as elemental B. The milling was done in isopropanol for 16 h, using WC-Co milling media with 1 wt.% PVP* added as a binder. Media wear during milling contributed ~0.3 wt.% WC-Co to each of the compositions. Following milling, both pre-alloyed intermetallics and the reaction sintered mixtures were dried and screened to -100 mesh. Specimens were uniaxially pressed in either 25 or 55 mm diameter steel dies at ~70 MPa (10 ksi) and isopressed at 350 MPa (50 ksi).

Batch sintering was done in a graphite element furnace at temperatures of 1450 or 1500°C. The heating schedule consisted of a ramp of 10°C/min from room temperature to 1200°C, a 0.5 h hold at

1200°C for degassing, and another ramp of 10°C/min to the final sintering temperature; all under vacuum. The temperature was maintained at the sintering temperature for 0.5 h under vacuum followed by an argon gas pressurization to 1 MPa (150 psi) in 10 min and a hold under pressure for 10 minutes. The total time at the sintering temperature was 50 min.

For continuous sintering, a Model 44BF belt furnace manufactured by Centorr Vacuum Industries, Inc., Nashua, NH, with a graphite hot zone was used. The specimens were loaded into graphite boats with lids and placed on the high-temperature SiC link belt. The furnace was brought up to operating temperature under flowing Ar (flow rate approximately 110 liters/min) and the belt speed was adjusted to 18.75 mm/min (0.75"/min). This produced an effective heating rate of about 6.5°C/min from room temperature to 300°C and 60°C from 300°C to the peak temperature of 1450 or 1500°C. The time at peak temperature was 50 min.

For all of the test samples, densities were determined by the Archimedes' method in distilled, de-ionized water. Specimens were then diamond polished and hardness and fracture toughness measurements made by diamond indentation, using 20 kg and 50 kg loads. Vickers hardness and indentation fracture toughness were determined by standard indentation methods.[18,19] Microstructural investigation were performed on polished samples by SEM.

Table III. Ni_3Al Alloy Compositions

Major Alloying Element	Binder Composition (atom%)	Substitution Site of Alloying Element
Baseline	$Ni_{3.00}Al_{1.00}$	None
Fe	$Ni_{2.85}Fe_{0.20}Al_{0.95}$	Both Ni and Al
Si	$Ni_{3.00}Si_{0.20}Al_{0.80}$	Al
Ti	$Ni_{3.00}Ti_{0.20}Al_{0.80}$	Al
Mo	$Ni_{3.00}Mo_{0.20}Al_{0.80}$	Al
W	$Ni_{3.00}W_{0.20}Al_{0.80}$	Al
Co	$Ni_{2.40}Co_{0.60}Al_{1.00}$	Ni
Cr	$Ni_{2.85}Cr_{0.20}Al_{0.95}$	Both Ni and Al
Zr	$Ni_{3.00}Zr_{0.20}Al_{0.80}$	Al

RESULTS AND DISCUSSION

The density, microhardness and fracture toughness results are given in Tables IV through VII and shown graphically in Figs. 1 through 6. The microstructures of some of the cermets containing pre-alloyed and reaction sintered alloys that were sintered by both methods are shown in Figs. 7 through 18.

Table IV. Sintering Results for Intermetallic-Bonded TiC at 1450°C for 50 min.

Intermetallic Phase	Continuous Furnace (SIUC)	Pressure Furnace (ORNL)
	% Target Density	
$Ni_{2.85}Fe_{0.20}Al_{0.95}$	98.2	98.2
$Ni_{2.85}Cr_{0.20}Al_{0.95}$	93.6	92.7
$Ni_{3.00}Si_{0.20}Al_{0.80}$	99.5	98.2
$Ni_{3.00}Ti_{0.20}Al_{0.80}$	99.9	98.9
$Ni_{3.00}Mo_{0.20}Al_{0.80}$	99.6	99.1
$Ni_{3.00}W_{0.20}Al_{0.80}$	93.9	98.7
$Ni_{2.40}Co_{0.60}Al_{1.00}$	97.3	98.8
$Ni_{3.00}Zr_{0.20}Al_{0.80}$	84.3	91.0

Table V. Sintering Results for Intermetallic-Bonded TiC at 1500°C for 50 min.

Intermetallic Phase	Continuous Furnace (SIUC)	Pressure Furnace (ORNL)
	% Target Density	
$Ni_{2.85}Fe_{0.20}Al_{0.95}$	99.2	98.7
$Ni_{2.85}Cr_{0.20}Al_{0.95}$	95.1	97.2
$Ni_{3.00}Si_{0.20}Al_{0.80}$	99.5	99.3
$Ni_{3.00}Ti_{0.20}Al_{0.80}$	99.1	99.8
$Ni_{3.00}Mo_{0.20}Al_{0.80}$	98.9	100.3
$Ni_{3.00}W_{0.20}Al_{0.80}$	96.1	94.0
$Ni_{2.40}Co_{0.60}Al_{1.00}$	97.5	106.1
$Ni_{3.00}Zr_{0.20}Al_{0.80}$	86.7	95.8

The results in Table IV and Fig. 1 show that very high densities were obtained by both batch pressure and continuous sintering at 1450°C for many of the alloying elements. At this sintering temperature, continuous sintering produced higher densities for Si, Ti and Mo alloying elements, while batch pressure sintering produced higher densities for W, Co, and Zr. The poorest densification for either sintering method resulted when the alloying modifier was Zr.

By increasing the sintering temperature to 1500°C (Table V and Fig. 2), many of the densities were improved. At this sintering temperature, continuous sintering produced high densities for Fe, Si, and Ti alloying elements, while batch pressure sintering produced significantly higher densities for Mo and Co

alloying elements. For batch sintering at this temperature, densities for Fe and W were significantly reduced.

Table VI. Comparison of Vickers Hardness for Intermetallic-Bonded TiC Sintered in Ar at 1450°C for 50 min in a Continuous Furnace (SIUC) and a Pressure Furnace (ORNL)

Intermetallic Phase	50 kg Load			
	Continuous Furnace		Pressure Furnace	
	Hardness (kg/mm^2)	K_{IC} ($MPa \cdot m^{1/2}$)	Hardness (kg/mm^2)	K_{IC} ($MPa \cdot m^{1/2}$)
$Ni_{2.85}Fe_{0.20}Al_{0.95}$	879	9.73	946	10.93
$Ni_{3.00}Si_{0.20}Al_{0.80}$	870	10.05	945	10.50
$Ni_{3.00}Ti_{0.20}Al_{0.80}$	956	10.63	952	10.60
$Ni_{3.00}Mo_{0.20}Al_{0.80}$	927	10.59	988	10.51
$Ni_{3.00}W_{0.20}Al_{0.80}$	597	9.61	1042	10.44
$Ni_{2.40}Co_{0.60}Al_{1.00}$	910	9.86	1054	9.76

Table VII. Comparison of Vickers Hardness for Intermetallic-Bonded TiC Sintered in Ar at 1500°C for 50 min in a Continuous Furnace (SIUC) and a Pressure Furnace (ORNL)

Intermetallic Phase	50 kg Load			
	Continuous Furnace SIUC		Pressure Furnace ORNL	
	Hardness (kg/mm^2)	K_{IC} ($MPa \cdot m^{1/2}$)	Hardness (kg/mm^2)	K_{IC} ($MPa \cdot m^{1/2}$)
$Ni_{2.85}Fe_{0.20}Al_{0.95}$	914	9.66	887	10.04
$Ni_{3.00}Si_{0.20}Al_{0.80}$	891	10.34	908	10.27
$Ni_{3.00}Ti_{0.20}Al_{0.80}$	906	10.29	930	10.54
$Ni_{3.00}Mo_{0.20}Al_{0.80}$	873	10.09	969	10.67
$Ni_{3.00}W_{0.20}Al_{0.80}$	663	9.79	999	10.63
$Ni_{2.40}Co_{0.60}Al_{1.00}$	905	9.95	1000	9.08

Because the densities were low for both the Cr and Zr alloying modified alloys, the cermets containing these alloying modifiers were not tested for hardness and fracture toughness. A comparison of

the results obtained for batch pressure and continuous sintering at 1450°C (Table VI, Fig. 3 and Fig. 5) show that in general batch sintering produced higher hardness and marginally higher toughness for most of the alloying modifiers. The exceptions were for Ti and Mo, where continuous sintering produced marginally higher toughness and similar hardness. The highest hardness was obtained for the W and Co alloying modifiers, while the highest toughness was obtained for Fe as the alloying modifier.

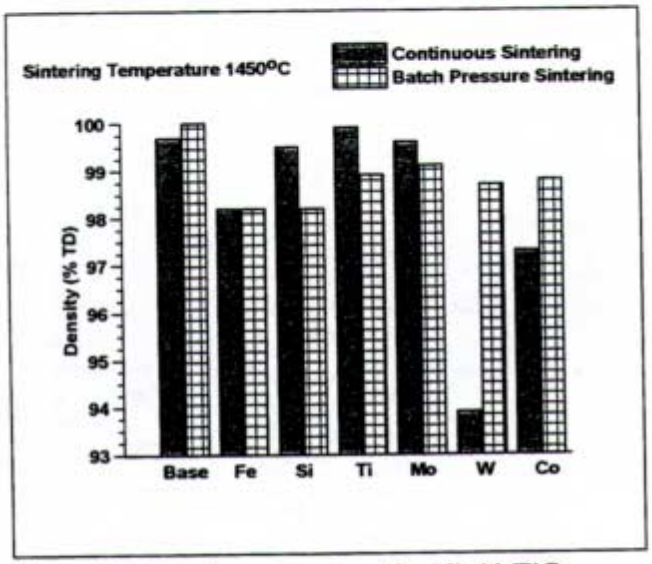

Figure 1. Density comparison for Ni_3Al-TiC with alloying additions batch pressure and continuous sintered in Ar at 1450°C.

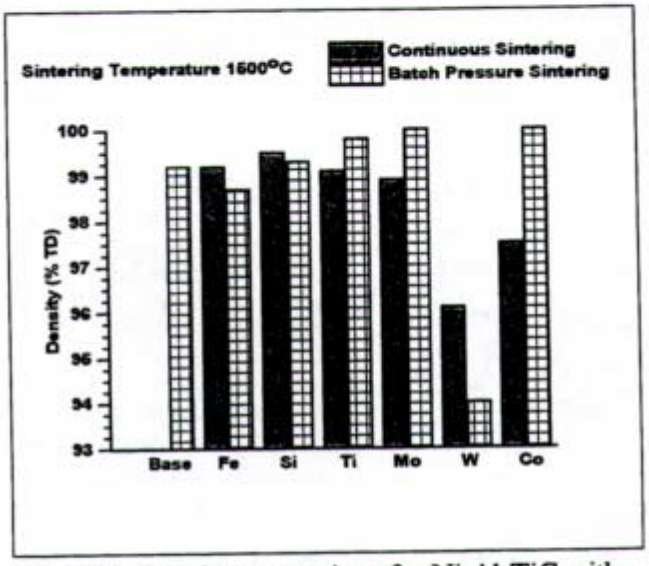

Figure 2. Density comparison for Ni_3Al-TiC with alloying additions batch pressure and continuous sintered in Ar at 1500°C.

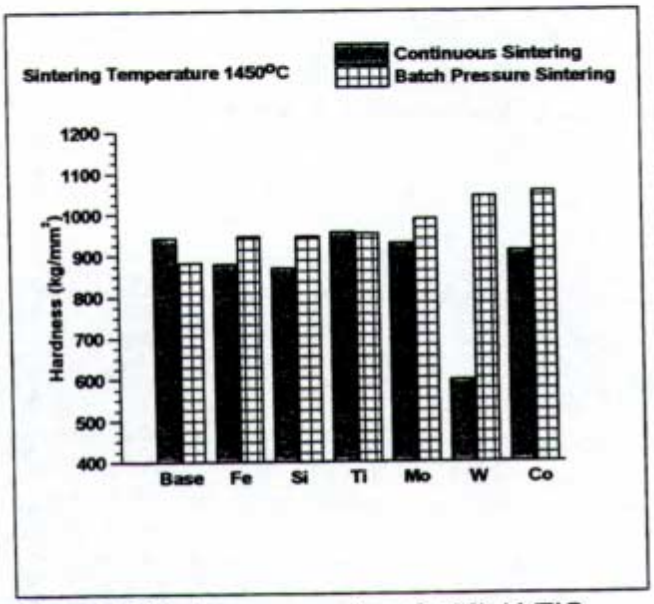

Figure 3. Hardness comparison for Ni_3Al-TiC with alloying additions batch pressure and continuous sintered in Ar at 1450°C.

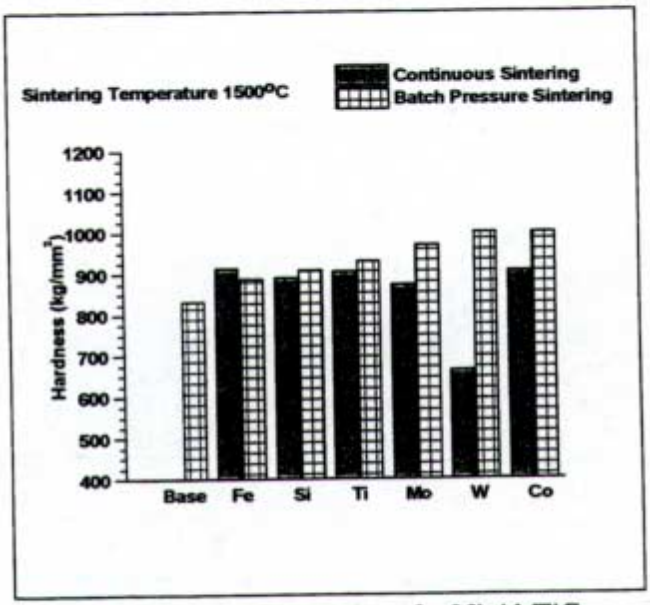

Figure 4. Hardness comparison for Ni_3Al-TiC with alloying additions batch pressure and continuous sintered in Ar at 1500°C.

For sintering at 1500°C (Table VII, Fig. 4 and Fig. 6), the hardness increased slightly for Fe and Si alloying modifiers for continuous sintering, while for batch pressure sintering the hardness for all alloying modifiers it slightly decreased. For Si and Ti similar hardness and toughness were obtained for both sintering methods, while significantly higher toughness resulted for the Co alloying modifier with continuous sintering.

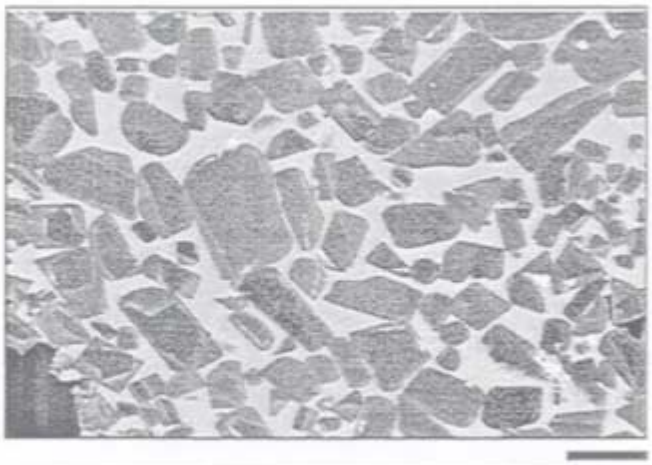

Figure 7. 30 vol.% Ni_3Al-TiC using pre-alloyed aluminide following batch pressure sintering at 1450°C. Bar = 5 μm.

Figure 8. 30 vol.% Ni_3Al-TiC using reaction sintered aluminide following batch pressure sintering at 1450°C. Bar = 5 μm.

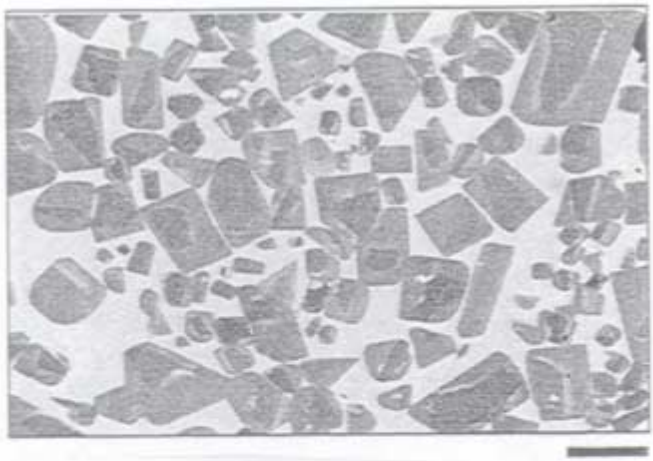

Figure 9. 30 vol.% Ni_3Al-TiC using pre-alloyed aluminide following batch pressure sintering at 1500°C. Bar = 5 μm.

Figure 10. 30 vol.% Ni_3Al-TiC using reaction sintered aluminide following batch pressure sintering at 1500°C. Bar = 5 μm.

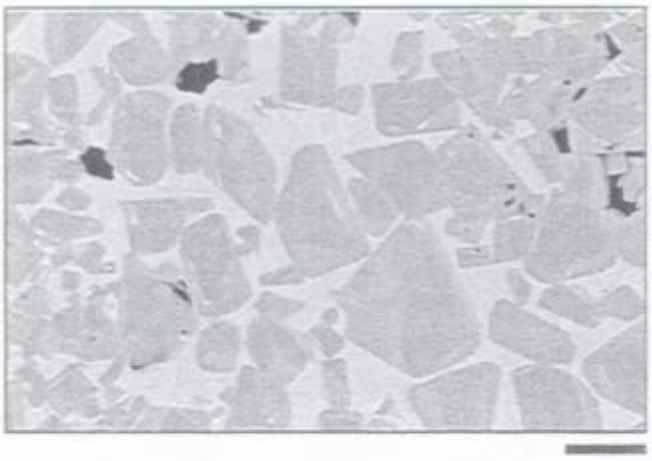

Figure 11. 30% $Ni_{2.85}Fe_{0.20}Al_{0.95}$ - TiC following batch pressure sintering at 1450°C. Bar = 5 μm.

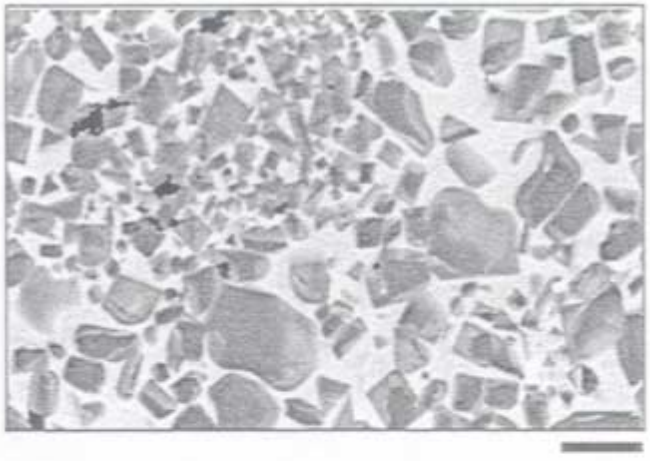

Figure 12. 30% $Ni_{2.85}Fe_{0.20}Al_{0.95}$ - TiC following continuous sintering at 1450°C. Bar = 5 μm.

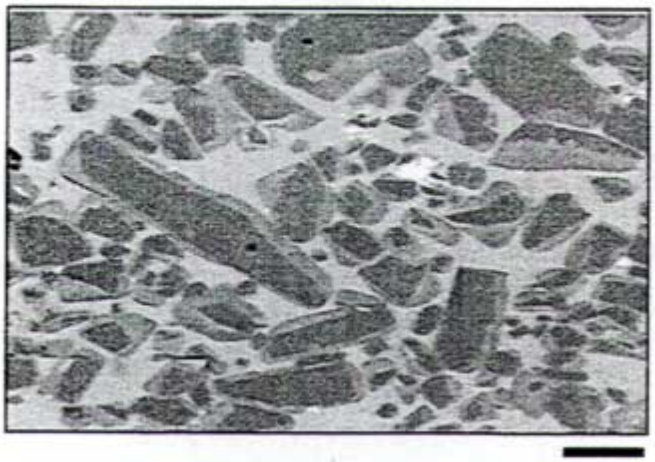

Figure 13. 30% $Ni_{3.00}$ $Mo_{0.20}$ $Al_{0.80}$ - TiC following batch pressure sintering at 1450°C. Bar = 5 μm.

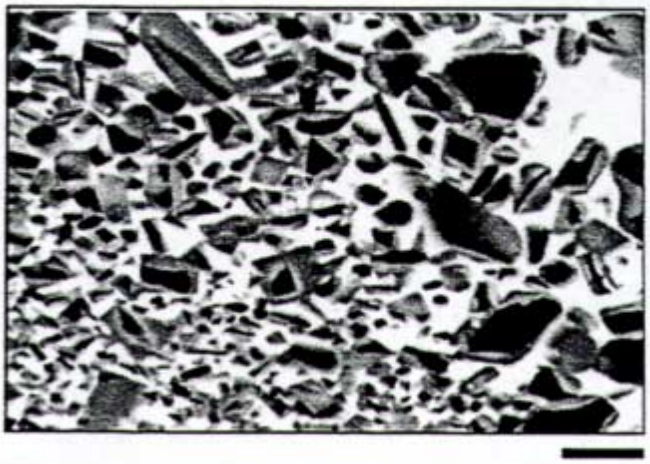

Figure 14. 30% $Ni_{3.00}$ $Mo_{0.20}$ $Al_{0.80}$ - TiC following continuous sintering at 1450°C. Bar = 5 μm.

Figure 15. 30% $Ni_{3.00}$ $Mo_{0.20}$ $Al_{0.80}$ - TiC following batch pressure sintering at 1500°C. Bar = 5 μm.

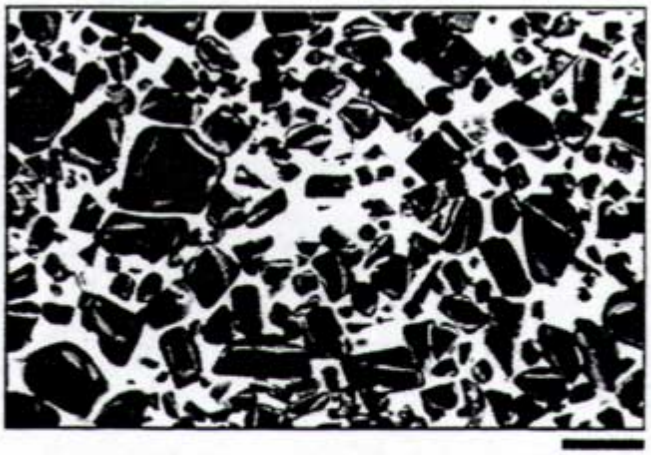

Figure 16. 30% $Ni_{3.00}$ $Mo_{0.20}$ $Al_{0.80}$ - TiC following continuous sintering at 1500°C. Bar = 5 μm.

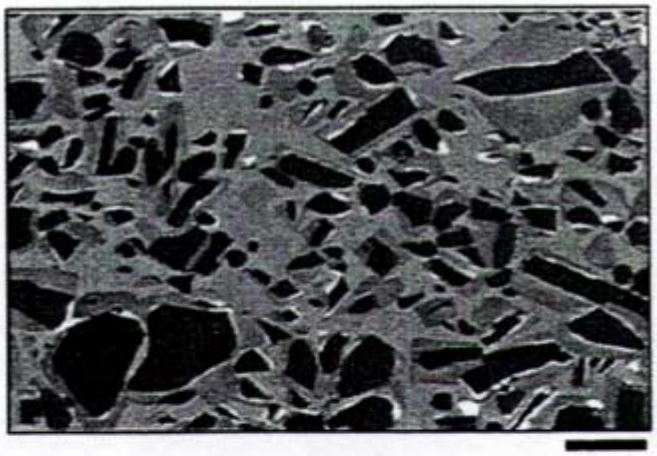

Figure 17. 30% $Ni_{3.00}$ $W_{0.20}$ $Al_{0.80}$ - TiC following batch pressure sintering at 1500°C. Bar = 5 μm.

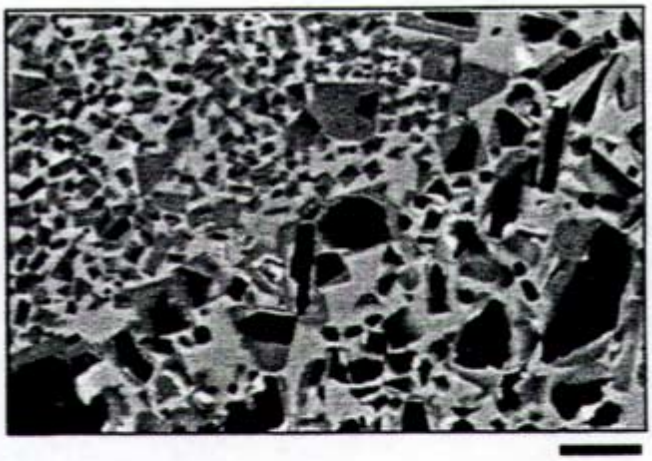

Figure 18. 30% $Ni_{3.00}$ $W_{0.20}$ $Al_{0.80}$ - TiC following continuous sintering at 1500°C. Bar = 5 μm.

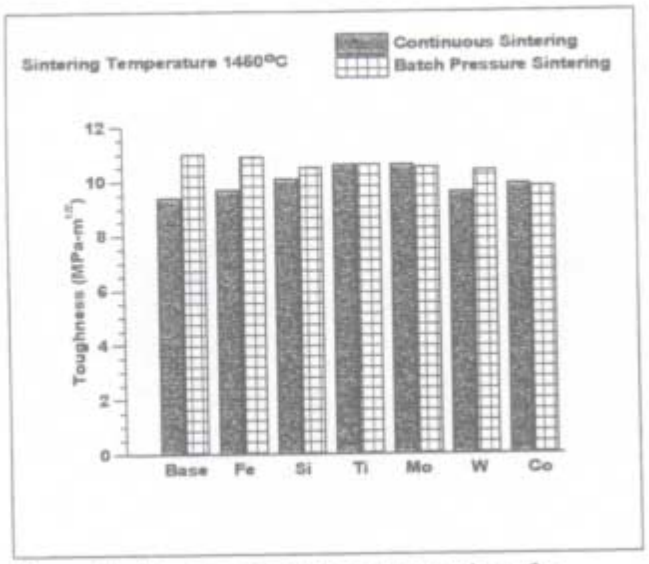

Figure 5. Fracture toughness comparison for Ni_3Al-TiC with alloying additions batch pressure and continuous sintered in Ar at 1450°C.

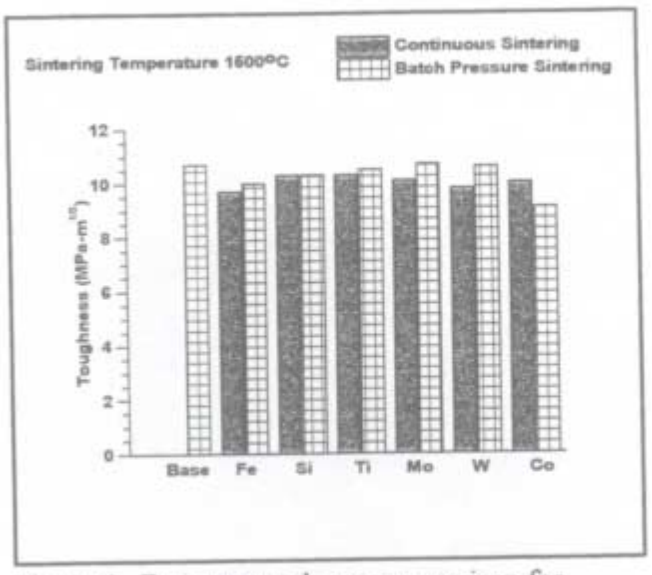

Figure 6. Fracture toughness comparison for Ni_3Al-TiC with alloying additions batch pressure and continuous sintered in Ar at 1500°C.

The microstructural results are given in Figs. 7 through 18. These photomicrographs show that for all cases considerable solubility of the TiC existed during reaction sintering in both the batch pressure furnace and the continuous furnace.

Figures 7 and 8 and 9 and 10, respectively, compare the microstructure produced by using pre-alloyed Ni_3Al and by forming Ni_3Al by reaction sintering. From previous work, it was shown that pre-alloying produces cermets with larger TiC grain sizes. This may be due to (1) enhanced reactivity due to the presence of more surface oxygen on the powders during reaction sintering; (2) enhanced diffusion of Ti in the liquid phase; or (3) a kinetic effect due to solution and reprecipitation of the TiC in the liquid phase. The photomicrographs (Figs. 7 and 9) show that the corners of the TiC have been rounded when using the using pre-alloyed Ni_3Al. This suggests that the points with highest radius of curvature were dissolved preferentially. In comparison, the cermets with Ni_3Al produced by reaction sintering (Figs. 8 and 10) show that the TiC surfaces appear to have been more uniformly reduced during sintering.

When Fe was used as an alloying element (Figs. 11 and 12), it would appear that continuous sintering produced more reprecipitated TiC than batch pressure sintering. However, the overall solubility of the TiC in the binder phase appears to be increased by the addition of Fe.

Similarly the addition of Mo and W as alloy modifiers (Figs. 13 through 18) appear to have had a major effect on the solubility of the TiC in the binder phase. Also, in general continuous sintering appears to have produced a finer TiC structure and a greater amount of reprecipitated TiC than batch pressure sintering.

CONCLUSIONS

This work showed that it is possible to use continuous sintering in flowing Ar as a means of producing dense cermets containing Ni_3Al alloys formed by reaction sintering. Continuous sintering was also found to have an effect on TiC grain size and solubility of TiC in the binder phase. Since the measured properties of the continuous sintered cermets were comparable to those of batch pressure sintered cermets of the same formulations, these results warrant additional investigation for further optimization of continuous sintering parameters.

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