MECHANICAL, THERMAL AND TRIBOLOGICAL PROPERTIES OF HOT-PRESSED NiAl/ Al_2O_3 COMPOSITES

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Abstract

This paper is concerned with the mechanical properties (bending strength, fracture toughness, hardness, Poisson ratio and Young modulus), thermal properties (thermal expansion coefficients, heat capacity, thermal conductivity) and tribological properties of the NiAl/Al_2O_3 composites. The composite materials were produced by the hot-pressed method using the NiAl/20(30)vol.%Al_2O_3 powder mixtures. The highest bending strength (635 MPa) was achieved in the NiAl/20vol.%Al_2O_3 composite and it was higher by about 80% than that of the 'pure' NiAl phase (345 MPa). Within the entire temperature range examined (from 50 to 800^oC) the thermal expansion coefficients of the composites were decidedly lower than that of the 'pure' NiAl phase. The values of the thermal expansion coefficients of the composite materials were: NiAl α=6.1-12.5x10^{-6}/^oC, NiAl/20vol.%Al_2O_3 α=5.5-11.1x10^{-6}/^oC and NiAl/30vol.%Al_2O_3 α=4.9-10.2x10^{-6}/^oC (as measured within the temperature range from 50 to 800^oC).

1 Introduction

Intermetallic phases of the Ni-Al type belong to the group of modern constructional materials which have low density and numerous excellent properties such as the high values of the melting temperature, resistance to high-temperature oxidation (to about 1200^{o}C), Young modulus (stable against temperature), mechanical strength, resistance to abrasion, fatigue strength, and tensile and compressive strengths (also at elevated temperatures) [1-10].

This unparalleled combination of unique physicochemical and mechanical properties of these materials offers great possibilities of their application in many industrial branches. They have already been used in technologically developed countries in such fields as e.g. the automobile, aircraft, spacecraft, metallurgical, chemical and power industries.

Intermetallic compounds have however also drawbacks in that they are quite brittle at room temperature which makes their mechanical processing very difficult and restricts their application range, and also in that their creep resistance at high temperatures is poor. These drawbacks can be obviated by modifying their chemical composition or subjecting them to a plastic and/or heat treatment.

Based on our preliminary studies and technological trials, and taking into account the literature reports, we can state that there is the possibility of producing materials with the advantages of intermetallic phases but without their drawbacks. This can be achieved in the composites with the matrix made of an intermetallic phase reinforced with ceramic.

This paper describes the mechanical properties (bending strength, fracture toughness, hardness, Poisson ratio and Young modulus), thermal properties (thermal expansion coefficients, heat capacity, thermal conductivity), and tribological properties of the NiAl/Al_2O_3 composites. The composite materials were obtained from the NiAl/20vol.%Al_2O_3 and NiAl/30vol.%Al_2O_3 powder mixtures by subjecting them to hot-pressing.

2 TECHNOLOGICAL EXPERIMENTS

The composite materials were produced by the hot-pressing method using the NiAl/20(30)vol.%Al_2O_3 powder mixtures. The NiAl/20(30)vol.%Al_2O_3 composites were sintered in an ASTRO HP50-7010 press using an argon protective atmosphere. The sintering parameters were: the sintering temperature T_s=1400^{o}C, sintering time T_s=30 min, pressure P~30 MPa. Fig.1 shows the microstructures of the composites thus obtained.

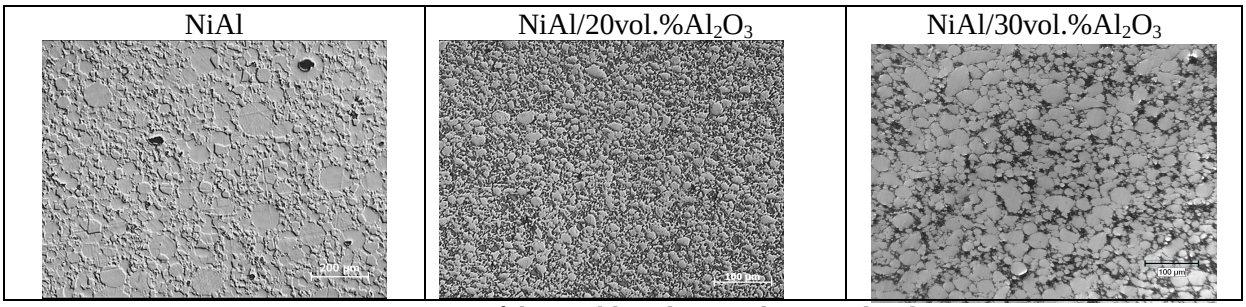


Figure.1. Microstructure of the NiAl-based materials sintered under pressure.

The sintered materials were subjected to examinations of their mechanical properties (bending strength, fracture toughness, hardness, Poisson's ratio, and Young modulus), thermal properties (thermal expansion coefficients, heat capacity, thermal conductivity) and tribological properties.

3. MECHANICAL PROPERTIES

The density was determined using the hydrostatic method. The bending strength, fracture toughness, were examined in a ZWICK 1446 strength machine at a support spacing of 40 mm and a head travel speed of 1.0 mm/min. The samples intended for the bending strength tests were sized at 5x5x50 mm, and the head load was 1kN.

The fracture toughness was also tested by three-point bending. The samples were notched beams sized at 5x5x50 mm. The notch was made in the two stages: the samples were first notched to a depth of 0.9 mm using a wheel 0.2 mm wide and, then, deepened to about 1.1 mm (the total depth of the notch) with a wheel 0.023 mm wide. The straining rate, i.e. the head travel speed was 1.0 mm/min and the head load was 10 kN.

The hardness of the composites was determined in a Vickers hardness-meter under a load of 10 kG applied for 10 s. Each sample was tested 5 times (five measurements).

The Young modulus and the Poisson ratio were examined using the pulse-echo ultrasonic technique. The velocities of the longitudinal V_L and transversal V_T ultrasonic waves (f= 5-10 MHz) were measured in cubicoidal samples (10x10x5 mm) with high accuracy (digital flaw detector + appropriate software). In isotropic elastic materials, 2 independent elastic constants should be taken into account. They can be calculated from the formulae (where V_L and V_T are the ultrasonic velocities and ρ is the mass density):

$$E = \rho \frac{(3V_L^2 V_T^2 - 4V_T^4)}{(V_L^2 - V_T^2)} \quad (1)$$

$$v = \frac{\left(\frac{1}{2}V_L^2 - V_T^2\right)}{(V_L^2 - V_T^2)}$$

Material [% vol.]	**Bending strength σ_c [MPa]**	**K_{IC} [MPa·m1/2]**	**Young modulus [GPa]**	**Poisson ratio ν**	**Hardness HV10 [GPa]**	**Measured density [g/cm^3]**	**Relative density [%]**
NiAl	345.6±53.8	7.2±0.4	188.0	-	3.08±0.1	5.88	99.5
NiAl/20Al_2O_3	635.2±43.7	6.6±0.2	216.3	0.289	4.29±0.5	5.51	99.9
NiAl/30Al_2O_3	456.2±37.0	7.4±0.3	208.3	0.253	5.61	5.30	99.4

(2)

Table 1. Properties of the hot-pressed NiAl/Al_2O_3 composite.

We can see from Table 1 that the bending strength of the NiAl/20vol%Al_2O_3 composite (635 MPa) is higher by 80% than that of the 'pure' NiAl phase (345 MPa), but the fracture toughness of the three materials differs however only slightly. The Young modulus of the composites is higher by about 12% than that of the 'pure' NiAl phase. The hardness of the composite exceeds that of NiAl by about 40%.

The addition of the ceramic phase to the NiAl matrix has an important effect on the course of failure of the composite material. This can be seen in Fig.2 which shows the microstructures of fractures of the sintered NiAl-based materials after bending tests. Characteristically, in the pure NiAl phase (Fig.2a), failure proceeds in one direction through the NiAl grains, whereas in the composite material (Fig.2b), the presence of the ceramic particles results in the crack often changing its way thereby making it longer, which in effect increases the strength of the material.

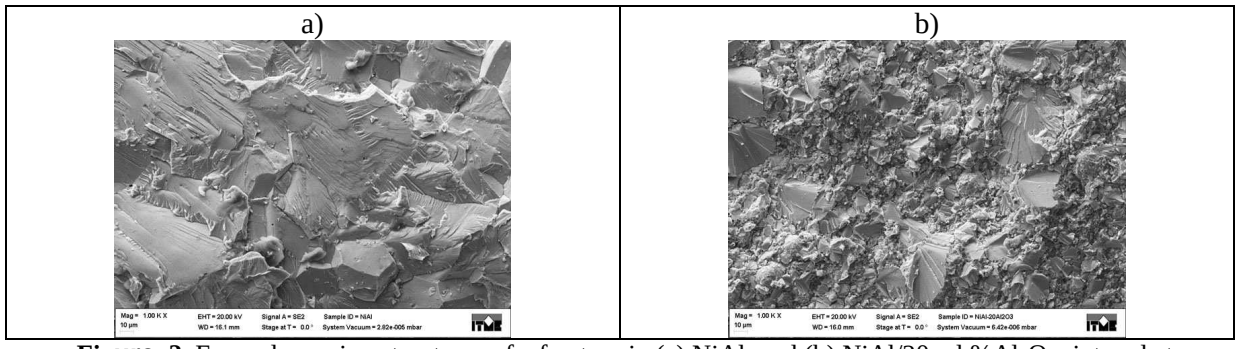


Figure. 2. Exemplary microstructures of a fracture in (a) NiAl, and (b) NiAl/20vol.%Al_2O_3 sintered at T_s=1400^{o}C, t_s=30 min, P~30 MPa.

4 THERMAL PROPERTIES

The thermal expansion coefficient was measured using a direct vertical dilatometer. The sample was heated to a temperature of 800^{o}C at a rate of 10^{o}C/min. This relatively high heating rate permitted minimizing the dilatation effects which may take place in the samples when they are placed in the furnace of the measuring device. After the samples were heated to 800^{o}C, they were cooled (still in the furnace) at a rate of 3^o/min. The results of the calculations are shown in Fig.3.

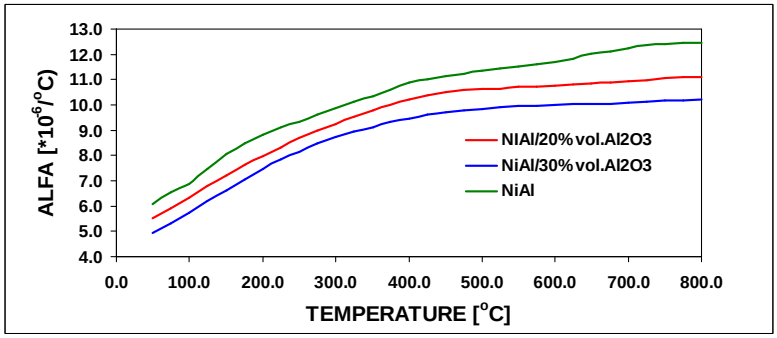


Figure. 3. Temperature dependence of the thermal expansion coefficient α in the NiAl/20(30)vol.%Al_2O_3 composite and 'pure' NiAl phase.

Within the entire temperature range examined (50 to 800°C) the thermal expansion coefficients of the composites are decidedly lower then than that of the 'pure' NiAl phase (NiAl α=6.1÷12.5x10^{-6}/°C, NiAl/20vol.%Al_2O_3 α=5,5÷11,1x10^{-6}/°C and NiAl/30vol.%Al_2O_3 α=4.9÷10.2x10^{-6}/°C). This is of crucial importance from the point of view of joining the composites with ceramic materials. During joining these two materials, the mismatch between their thermal coefficients results in adverse thermal residual stresses being generated (tensile in the ceramic). The most dangerous situation for ceramic-metal joints is when the tensile residual stresses active in the ceramic component add to the tensile exploitation stresses, so that their sum exceeds the strength of this component. This may lead to failure of the entire joint.

The heat capacity Cp of the composites was determined using an STA 449 F1 Jupiter simultaneous thermal analyzer (manufactured by the Netsch Co) and a differential scanning calorimeter (of the heat flow type). The temperature program and the registration of the heat flow between the crucible with the sample examined and the reference crucible were computer-controlled using the PROTEUS E SOFTWARE 5.1.0 (Netzch). The heat capacity of the materials was determined by comparing it with that of a sapphire reference (the ratio method) using the formula (3):

$$Cp = \frac{m_{standard}}{m_{sample}} * \frac{DSC_{sample} - DSC_{bas}}{DSC_{standard} - DSC_{bas}} * Cp_{standard} \qquad (3)$$

Where: Cp – heat capacity of the sample material at the temperature T, $Cp_{standard}$ – heat capacity of the reference, $m_{standard}$ – mass of the reference, m_{sample} – sample mass, $DSC_{standard}$ – magnitude of the DSC signal obtained for the reference at the temperature T, DSC_{sample} – magnitude of the DSC signal obtained for the sample at the temperature T, DSC_{base} – magnitude of the DSC signal for the base line at the temperature T

The calculated values of the heat capacity of the NiAl/20(30)vol%Al_2O_3 composites are shown in Fig.4.

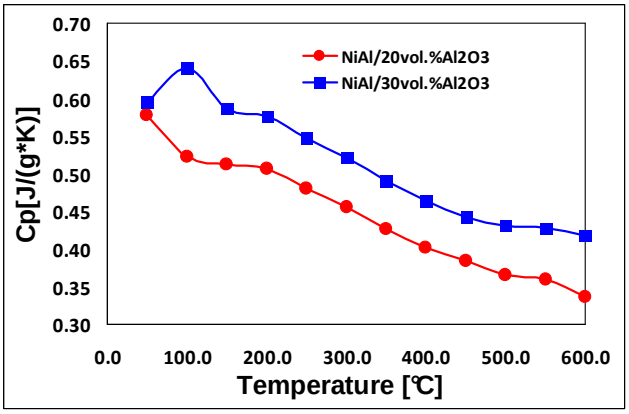


Figure. 4. Temperature dependence of the heat capacity Cp in the NiAl/20(30)vol.%Al_2O_3 composite.

The thermal conductivity λ was determined using an indirect method i.e. by measuring the thermal diffusivity c_p and the density ρ (as time-dependent quantities) and then calculating the thermal conductivity from the equation

$$\lambda(T) = a(T) \cdot \rho(T) \cdot c_p(T) \tag{4}$$

The thermal conductivity of the composites was determined within the temperature range from 50 to 400°C (the intermediated measurements were made at 100, 200, and 300°C). The thermal diffusivity was measured using a mathematical model based on the CAPE-LEHMAN non-linear regression with pulse correction, which takes into account the radiation and facial heat losses). The values of the thermal conductivity λ of the NiAl/20(30)vol.%Al_2O_3 composites determined in this way are shown in Fig.5 and Table 2.

Temperature [°C]	**Thermal conductivity [W/m*K]**	
	NiAl/20vol.%Al_2O_3	**NiAl/30vol.%Al_2O_3**
50	58.61	53.63
100	50.72	54.30
200	46.66	43.14
400	32.79	30.11
600	25.08	24.50

Table 2. Thermal conductivity of the NiAl/20(30)vol.%Al_2O_3 composite.

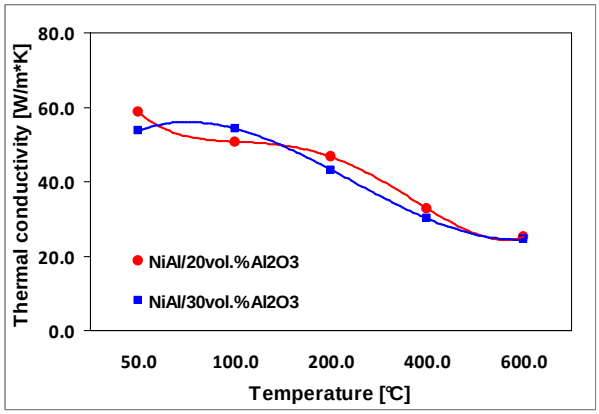


Figure. 5. Thermal conductivity of the NiAl/20(30)vol.%Al_2O_3 composite.

5. TRIBOLOGICAL PROPERTIES

The frictional wear resistance was measured by the ball-plane using a DUCOM tribo-tester The samples were pressed against a sapphire ball, 6.5mm in diameter, with the force F_n=15 N. The holder together with the ball fixed in it was set in the reciprocating motion driven by an electro-dynamic generator. The two components in friction slid upon one another at a velocity of 5 mm/s in periods of 15.0min. The friction force F_t was recorded with a piezoelectric displacement sensor, the measurements being taken 24 times per second. After the test, the wear resistance of the samples was measured with a scanning profile-gauge (Form Talysurf Series 2).

The results of the measurements (Table 3, Fig.6) included a 3D image of the sample wear, the volume of the groove (representing the wear size), and the wear profile in the direction perpendicular to the groove axis. The measurements were taken along 1mm of the groove length. Fig.6 shows an example of the isometric map of the sample surface and a transverse cross-section of the wear profile

Material	**Volume [$\times 10^6$ μm^3]**
NiAl	76.7
NiAl/20vol.%Al_2O_3	30.4
NiAl/30vol.%Al_2O_3	20.7

Table 3. The volume of the groove area after wear test.

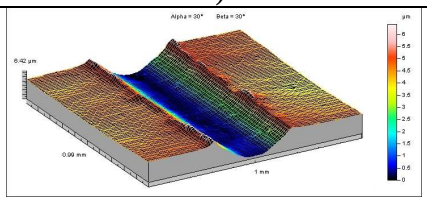
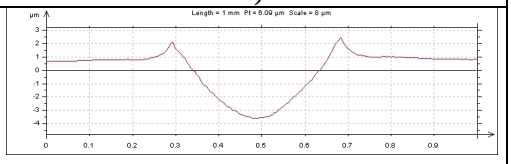
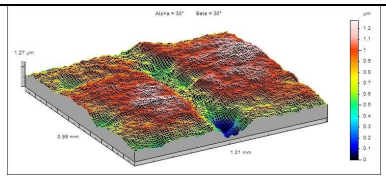
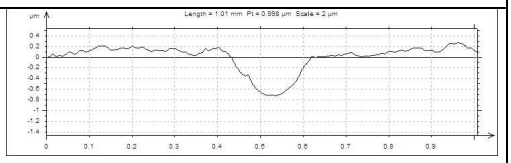
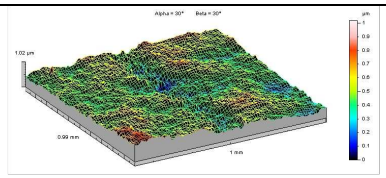
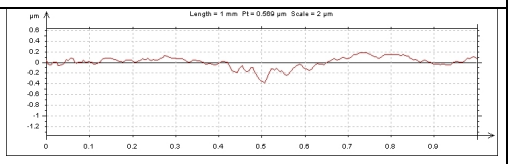
Material	a)	b)
NiAl		
NiAl/20vol.%Al_2O_3		
NiAl/30vol.%Al_2O_3		

Figure. 6. Wear profiles obtained for NiAl/20(30)vol.%Al_2O_3 composite materials.

As follows from Table 2 and Fig.6, the wear resistance of the composites is excellent. Compared to that measured in the pure NiAl phase, the volume of the wear groove is halved in the NiAl/20vol%Al_2O_3 composite and less than one third in the NiAl/30vol%Al_2O_3 composite.

The friction coefficient in the NiAl/20(30)vol.%Al_2O_3 composite and 'pure' NiAl phase was determined from the relationship:

$$\mu = F_t/F_n \qquad (4)$$

where: μ - the friction coefficient, F_t –the friction force, and F_n – the load

Fig.6 shows plots of the averaged values of the friction coefficient determined for the NiAl/20(30)vol.%Al_2O_3 composites and 'pure' NiAl phase. In all the three materials the friction coefficient initially increases considerably and, then, it stabilizes for a short time (about 200s). . Afterwards, it considerably increases to stabilize at a value of about 0.85 in pure NiAl, whereas markedly decreases in the NiAl/Al_2O_3 composites to finally stabilize at about 0.4 in the NiAl/30vol.%Al_2O_3 composite and at 0.35 in the NiAl/20vol.%Al_2O_3 composite.

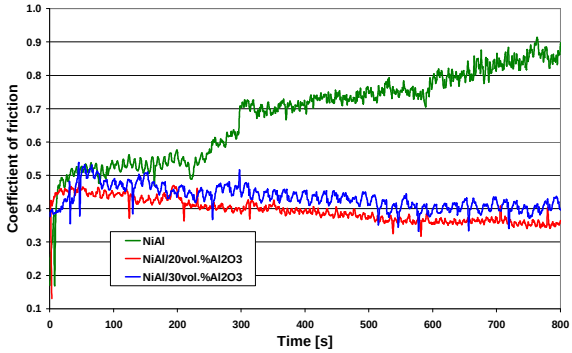


Figure. 6. Variation of the friction coefficient in the NiAl/20(30)vol.%Al_2O_3 composites and 'pure' NiAl phase.

6. SUMMARY

The mechanical properties (bending strength, fracture toughness, hardness, Poisson ratio and Young modulus), thermal properties (thermal expansion coefficients, thermal conductivity) and tribological properties of the composites with the matrix made of the Ni-Al intermetallic material reinforced with the Al_2O_3 ceramic phases were examined in dependence on the content of the reinforcing phase. The composite materials were produced from the NiAl/20vol.%Al_2O_3 and NiAl/30vol.%Al_2O_3 powder mixtures by subjecting them to hot-pressing

The bending strength of the composites thus produced appeared to be higher than that of the pure Ni-Al phase (345 MPa) by about 80% in the NiAl/20vol.%Al_2O_3 composite (635 MPa) and by about 32% in the NiAl30vol.%Al_2O_3 composite (456 MPa), but the fracture toughness of the composites was not much improved compared to that of the pure NiAl phase. The hardness of the composites increases with increasing ceramic content. Compared to that of NIAl (3.1 GPa) it is improved by about 40% in the NiAl/20vol./%Al_2O_3 composite (4.3 GPa) and by about 80% in the NiAl/30vol.%Al_2O_3 composite (5.6 GPa).

The presence of the ceramic phase and the refinement of the NiAl powder grains affect essentially the failure process of the composite samples. In the presence of the hard reinforcing grains the cracks do not run through the NiAl grains but wind their way between the ceramic grains. In effect the cracking route is greatly elongated and the bending strength increases.

Within the entire temperature range examined (50 to 800^{o}C) the thermal expansion coefficients of the composites are decidedly lower then than that of the 'pure' NiAl phase.

Their values obtained in the present experiments are: NiAl α=6.1÷12.5x10^{-6}/^{o}C, NiAl/20vol.%Al_2O_3 α=5,5÷11,1x10^{-6}/^{o}C and NiAl/30vol.%Al_2O_3 α=4.9÷10.2x10^{-6}/^{o}C.

The friction coefficients measured in the NiAl/Al_2O_3 composites were stable and well reproducible. Their final values were 0.35 in the NiAl/20vol.%Al_2O_3 composite and 0.4 in the NiAl/30vol.%Al_2O_3 composite. The main advantage of the NiAl/Al_2O_3 composite was their increased frictional wear resistance with respect to that of the 'pure' NiAl phase. Compared to that measured in the pure NiAl phase, the volume of the wear groove (representing the wear size) was halved in the NiAl/20vol%Al_2O_3 composite and less than one third in the NiAl/30vol%Al_2O_3 composite.

ACKNOLEDGMENTS

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