Effect of M_7C_3 nano particulates on wear resistance of Ni based MMCs

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Abstract: *The metal matrix composites of Ni-Al-M_7C_3 were fabricated by mechanical alloying process (1-4 h). Pure nickel, aluminum and FeCrC powders were mixed, mechanical alloyed and sintered in an atmosphere controlled furnace for 800-1100 ^oC temperature and 1-3 hr time. The synthesis of the FeCrC added NiAl was attempted to improve the wear resistance of the Ni based MMCs composite. The initially added FeCrC particles were unstable and decomposed partially within the matrix during sintering, and the carbides formed as nano sized. The microhardnes, XRD, SEM, EDS, optical micrographs and dry sliding wear mechanism of the samples were investigated.It was confirmed that the mechanical alloying time decreased sintering time, formed nano sized reinforcements and improved the wear resistance of Ni based M_7C_3 reinforced MMCs.*

Key Words: *Ni based alloys, nano reinforcement, mechanical alloying*

1. Introduction

Mechanical alloying, represents an attractive technique for the preparation of intermetallics and a variety of other materials, including carbides, borides, nitrides, silicides, hydrides, composites, etc. [1-7]. Depending on the alloying conditions, two entirely different reaction kinetics are possible; the reaction may extend to a very small volume during each collision, resulting in a gradual transformation, or if the reaction enthalpy is sufficiently high, a self-propagating combustion reaction can be initiated. The volume combustion conducts a gradual heating of the sample uniformly in a controlled atmosphere until the reaction occurs simultaneously throughout the entire sample. Temperatures of the reactions relate to the reaction synthesis, compaction of the raw materials, specific surface, and other processing parameters. The procedure has low energy consumption and has large potentials for industrial applications due to the high productivity and the simplicity of the equipment involved. In addition, synthesis and sintering could be made within only one-step [3]. Due to high temperatures and velocities of reactions, limited information is available on the mechanism and on the kinetics of these processes. Intermetallic compounds and IMCs are produced commercially by conventional manufacturing methods. However, the method based on the principle of sintering synthesis appears to be promising for ease of production [8-12]. Studies on the sintering synthesis of intermetallic compounds have focused to a large extent on the NiAl and Ni_3Al composites [9]. Intermetallic compounds, especially metal aluminides (such as nickel, titanium, cobalt, and niobium aluminides) have been considered as promising materials for high-temperature structural applications [10]. This is due to their physical and mechanical properties, which include low density, high specific strength, excellent creep resistance, and good oxidation and corrosion resistance. Conventional processing techniques used to fabricate intermetallic compounds are generally through a combination of melting, casting, powder grinding, and consolidation by hot pressing. However, these techniques such as melting and casting methods are inapplicable to the fabrication of many intermetallic alloys due to, for example, a large difference between the melting points of constituent [11-14]. Nickel aluminized intermetallic compounds (NiAl and Ni_3Al) have received considerable attention for high temperature structural and coating applications, for example, as heat shields for sintering chambers and as vanes in industrial gas turbines [13]. The interface of Ni- M_7C_3 particulates is thermodynamically unstable [1]. Hence, the sintering temperature is very important on dissolution rate of M_7C_3 in Ni matrix. Some studies were performed on the positive effects of the interfacial reactions on the mechanical properties of the Ni-carbide composites [4], and various studies were performed on the high-temperature strength of the Ni based composites reinforced by continuous or discontinuous ceramic particulates [15,16]. In the present work, mechanical alloying was applied for the fabrication of M_7C_3 added Ni_3Al IMCs, and the objective is to

investigate the effect of mechanical alloying on decomposition and distribution rate of initially added M_7C_3 particles on the Ni_3Al IMCs.

2. Experimental procedure

Commercial nickel (99% purity, 100 μm average particle size), aluminum (99.8% purity, 100 μm average particle size) and M_7C_3 (99% purity, 100 μm average particle size) were used. The base powder compositions were prepared from 86.71 wt.%Ni and 13.28 wt.%Al. M_7C_3. Powder was mixed with the base powder at the ratios of 10 wt.% (Table 1).

Table 1. Production rough of samples

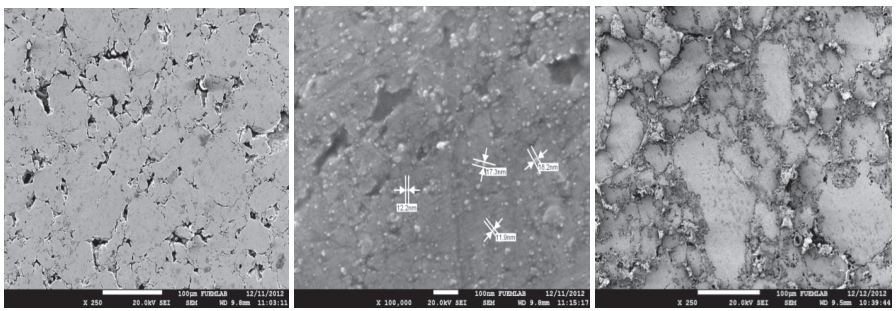
Sample	Mechanical alloying Time, (h)	Sintering Temperature, (^oC)	Sintering Time, (h)	Reinforcement, (wt.%)
S1.1	1	800	1	10
S1.2	1	800	2	10
S2.1	1	900	1	0
S2.2	1	900	1	5
S2.3	1	900	1	10
S2.4	1	900	1	20
S2.5	1	900	1	30
S3.2	1	900	2	10
S4.3	1	1000	2	10
S5.2	1	1100	2	10
S7.2	2	1000	2	10
S8.2	4	1000	2	10

The powder compositions were prepared as represented in Table 1. The prepared samples were mechanical alloyed in a spex type atritor for 1, 2 and 4 hr. The milled powder mixtures were pressed to form a pellet in a cylindrical mould under a uniaxial pressure (300 MPa). Subsequently, the compacts were placed into a vacuum tubular furnace and the sintering were carried out at 800-1100 ^oC for 1 and 2 hr. Microstructural evolution of the samples was investigated by optical and scanning electron microscope (SEM). Elemental analysis was carried out by using Tracor-Northern brand energy dispersive X-ray analyzer (EDX) attached to the SEM. Information about the variations of the hardness of the intermetallic phases were obtained by Vickers microhardness method ($HV_{0.2}$), and the phase vol.% were determined by image analyzer. Phase analysis was carried out by the X-Ray Diffraction (XRD) via CuK_α.

3. Results and Discussion

The production parameters of compact samples are listed in Table 1. During sintering, the exothermic reaction between nickel and aluminum powders generates heat. This heat provides a faster diffusion than encountered in the solid state leading to enhanced densification. The type and the amount of phases formed depends on the MA time, reinforcement wt.% and sintering temperature [15]. The SEM micrographs of sample $S_{1.2}$ was shown in Fig. 1.a-c, respectively. M_7C_3 was added to the samples S_1 group as 10 wt.%. It is seen from SEM micrographs that carbides and intermetallics were distributed homogeneously as particulate form in microstructure (Fig. 1.b). The presence of M_7C_3 carbide changed the shape of intermetallics (Fig 1.c). From XRD diagrams, it can be seen that due to the presence of chromium carbide as reinforcement intermetallic phases were impeded (Fig. 2.a-c). Ni, Ni_3Al, NiCrFe, NiAl, Cr_7C_3, and $Cr_{23}C_6$ phases are present and the matrix phase is α-Ni phase having Ni_3Al intermetallics (Fig. 1.b-c). From Fig. 2.a it is seen that the increase of the reinforcement depressed formation of intermetallics. XRD results of the samples S_2 and S_4 were given in Fig.2a, respectively. MA of Ni, Al and M_7C_3 mixture together eliminated Ni and Al and formed NiCrFe solid solution phase having a microhardness of approximately 400-150 HV. In this structure, also some locations having Ni_3Al and

chromium show a microhardness of 500-800 HV hardness. The MA time increased the Ni_3Al concentration and additionally $Cr_{23}C_6$ carbides were formed after 1h milling (Fig. 2). Sintering temperature increased Ni_3Al concentration, but after 1000 ^oC temperature $M_{23}C_6$ carbides formed, which is unwanted carbide type. It is seen from the figures that the samples contain especially Ni, Ni_3Al, NiAl and M_7C_3 phases and other minor phases (Fig. 2). It was detected from the metallographic examination and the XRD results that NiAl phase ratio is approximately 80-85 vol.% for the samples having M_7C_3 reinforcement. Stochiometric Ni-Al composition can produce as much as 50 vol.% liquid phase when the sintering temperature is in the range of 600-700 °C [16], hence the sintering temperature was increased gradually from 600 to 1100 °C. It can be seen that the size of secondary phases at the grain boundaries of the sample $S_{4.3}$ were increased. M_7C_3 particles were dissolved due to the nature of the mechanical alloying process which exhibits exothermic reaction, and the temperature of the exothermic reaction reached to the dissolution temperature of the M_7C_3 reinforce particulates. The size of secondary phase at the boundary of the crystallite and the pores increased (Fig. 3), and the size of the grains decreased by the increase of the sintering temperature. The increase of the M_7C_3 carbide reinforce ratio changed the dissolution rate and reactive reaction temperature. If the sintering temperature reaches the melting point of the aluminates and more, the evolution and entrapment of low melting point impurities or gaseous phase forms during the violent reactions.



(a) (b) (c)

Fig. 1. SEM micrograph of sample $S_{1.2}$

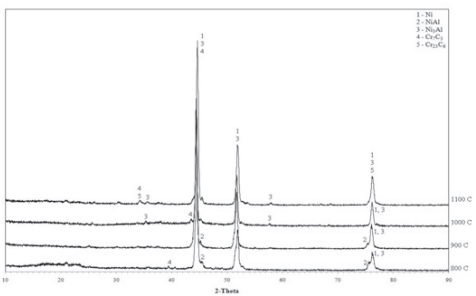


Fig. 2. XRD patterns obtained for the samples obtained as $S_{1.2}$, $S_{3\text{-}2}$, $S_{4.3}$, $S_{5.2}$

The sintering reactions involve definite interaction of solid Ni with Al-rich liquid. Reaction diffusion model is the one that is most commonly used to describe this kind of interaction. In that case, the product layer grows due to mass transport and the growth is

unrelated to the dissolution of the layer. An example of this can be found in isothermal dissolution of Ni in unsaturated liquid Al where intermetallic layer, Al_3Ni forms up to a temperature of 800 ^oC [16]. The first layer that forms due to the solid-liquid interaction contains Al_3Ni. Simultaneously, the temperature also increases continuously and leads to progressive dissolution of the layer. When it reaches 800 ^oC, Al_3Ni starts melting. The Al_3Ni layer, which is now in contact with the Ni particle, gives rise to phases richer in Ni. More importantly, microstructures have revealed one common reaction mechanism that is operative in mechanical alloying of Ni-Al powders [17].

The variation of the wear rate of samples with sliding distance are interpreted in Figure 3a. After 1200 m sliding distance asteady state was obtained, and the lowest wear rate was obtaned for the sample sintered at 1100 ^oC. The effect of sintering time can be seen from the Figure 2, and the effect of mechanical alloying on wear rate is labelled on figure 4b. The best mechanical alloying time can be seen as 2 h (Figure 3c). The increase of the mechanical alloying time to 4 h produced a sample having higher wear rate. Additionally, the effect reinforcement on wear rate was investigated on the figüre 4d, where it is seen that the best reinforcement ratio is 20 wt.%.

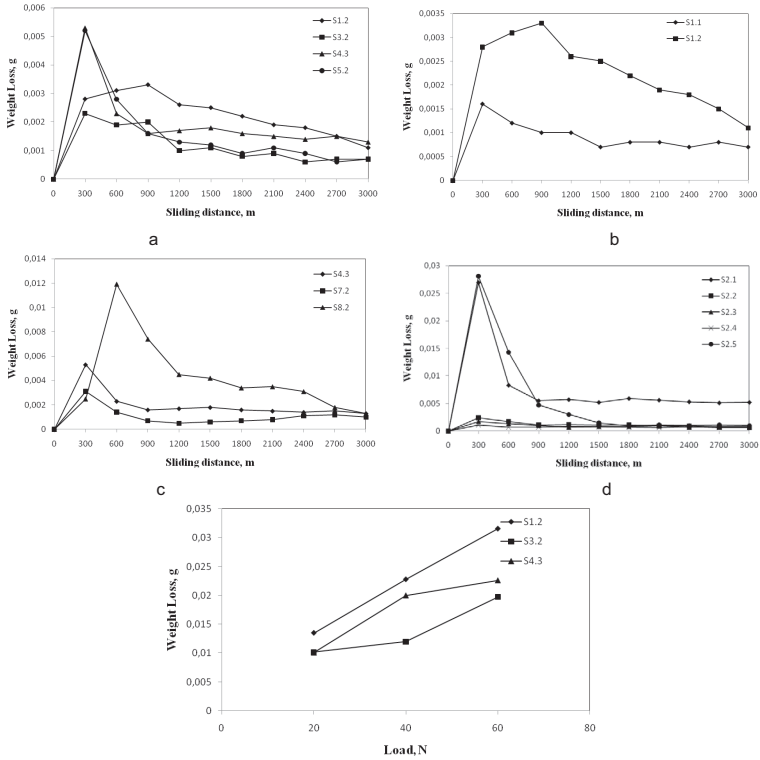


Fig. 3. a) Wear rate of the samples as function of sintering temperature, b) The effect of sintering time on the wear rate of the samples, c) The effect of mechanical alloying on the wear rate of the samples, d) The effect of reinforcement ratio on wear rate, e)The effect of the load on the wear rate

Figure 3e. shows the effect of the load and sliding distance on the wear rate. The wear rates of the surfaces, which also can be renamed as IMC composite, increase

linearly through the entire applied load range. The wear rate curves show that the change of wear rate with sliding distance is in the form of polynomial function. On the Figure 3, these functions were marked. From the Figure 3a, it was seen that, after a certain sliding distance and at constant velocity a transition to mild wear regime have been determined. Actually, this transition occurs when a critical temperature is reached at the contact surface, and this is observed when the critical temperature is reached at the contact surface as a result of frictional heating. The wear resistance of the composites is clearly demonstrated in the figures from the beginning of the test. A steady state value of wear rate seemed to be attained for all materials after sliding of 800 m, and the composite properties are still better than those of the reference sample. Figure 3e shows the amount of wear for different surface treated specimens as a function of the test load and the wear increases with the test load for all surface treated samples Results indicate that mechanical alloying improve the wear resistance of the samples compared to the reference sample. This becomes more apparent as the test conditions become increasingly severe. The surface hardness of the samples were increased from 100 HV to 250 HV by the increase of the reinforcement ratio, which is thought to be also due to new intermetallic phases. The increase of intrermetallic phase volume in the structure decreased the wear rate in considerable amount. The variation of the microhardness of samples with wear rate is also interpreted in Figure 3c. The hardness profiles demonstrate that significant surface hardening has been achieved by mechanical alloying. The results demonstrate that the wear rate of the samples are the most sensitive against carbide vol. %. These results show that the mechanical and tribological properties of the sampless depend strongly on the type of reinforcement phase, grain size, distribution of carbides and intermetallic phases, and alloying elemental concentration. Since the reinforcements, MC, M_7C_3 carbides, are relatively harder than matrix phases, α ferrite, austenite (γ). The mechanical alloying processed samples exhibits significantly higher hardness values compared to those obtained with conventional techniques due to the fairly uniform distribution of hard carbides in both the primary phase and the eutectic and the presence of a highly strained austenite (γ) phase.

4. Conclusion

Depending on the sintering temperature, the grain size of the composites decreased up to 1000 ^oC. Over this temperature gain boundary intermetallics were observed. The increase of the M_7C_3 carbide reinforce particulates ratio changes the dissolution rate and reactive reaction temperature. The melting of the Al-rich phase initiates the sintering of the new formed microstructure, and the sintering reactions involve definite interaction of solid Ni with Al-rich liquid. During sintering and dissolution of M_7C_3, Cr, Fe and C atoms dissolved to all of the phases, and the dissolution of these atoms changed the microstructure. Depending on Cr concentration, the dissolution temperature of Al_3Ni phase was increased, and Al_3Ni_2 phase was not seen in the structure. The increase of the M_7C_3 reinforce ratio increases Ni_3Al phase vol.%. The intergranular phase is NiAl, and the matrix phase is Ni_3Al. The size of secondary phase at the grain boundaries and the pores increased and the size of the grains decreased with sintering temperature. The weight loss of the samples increase linearly through the entire applied load range. The best production parameters were detected as 900 ^oC sintering temperature, 1h sintering time, 20 wt.% reinforcement ratio, and 2h mechanical alloying time.

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