



THE EFFECT OF COMPACTION PRESSURES ON THE MICROSTRUCTURE AND PROPERTIES OF NiAl/Ti FORMED BY SHS PROCESS

Tri Widodo Besar Riyadi, Sarjito and Patna Partono

Faculty of Engineering, Universitas Muhammadiyah Surakarta, Jl. A. Yani Tromol Pos 1 Pabelan Kartasura Surakarta, Indonesia

E-Mail: Tri. Riyadi@ums.ac.id

ABSTRACT

In the SHS process, compaction pressure on the reactant pellet is an important part influencing the thermal conductivity of the reactant. The thermal conductivity of the reactant affects the heat propagation and the heat loss during the ignition and wave propagation of the reaction. The objective of this work was to study the effect of compaction pressure on the microstructure and properties of the synthesized product. A Ni/Al mixture and Ti layer were compacted to produce 100 MPa, 150 MPa and 200 MPa on the pellets and ignited to initiate the SHS process. The microstructure of the synthesized product was observed using XRD and SEM, whereas the properties of the synthesized products were evaluated using a Vickers microhardness tester. Microstructure analysis indicated that several intermetallic phases have existed in the bilayer product as a result of reactions between Ni/Al and Ti. An increase in the compaction pressure led to an increase in the formation of pores in the synthesized product. The microhardness of product fluctuated with an increase of the compaction pressure.

Keywords: compaction pressure, NiAl/Ti, microstructure, properties.

INTRODUCTION

High temperature application of machine component requires advance protection coating which has high oxidation, corrosion and wear resistances. Intermetallics have great potential for application in elevated temperature since they have high melting point, low density, and high oxidation and corrosion resistance. NiAl is one of intermetallics which has shown promising candidate to be applied in high temperature application such as protective coating since it has several advantageous such as high melting point, high hardness, and good oxidation and corrosion resistance (Stolo *et al.* 2000). The intermetallics of titanium aluminide systems such as Ti3Al and TiAl have also offered greater potential for high temperature application with low density, high specific strength, high specific stiffness, high oxidation resistance compared than conventional titanium alloys (Vaucher *et al.* 2011).

During the last four decades, self-propagation high temperature synthesis (SHS) is attractive to produce intermetallic system. In the present work, SHS process was used to synthesize and fabricate coating in one processing route. However, many literatures have reported that high combustion temperature of SHS process generates thermal shock during the synthesis process since it produces high thermal stress at the interface between coating and the substrate that leads to the detachment of coating from the substrate (Hong *et al.* 2010). Recently, the problem of adhesion between the substrate and the coating formed by SHS process has been significantly overcome using the application of underlayer (Riyadi, Zhang, Marchant, *et al.* 2014)(Riyadi, Zhang & Sarjito 2014). Since the urgency of underlayer was mainly to improve the adhesion strength, it is therefore required to melt and make a mutual diffusion at the interface between

coating and the steel substrate (Jadoon *et al.* 2004). Due to the melting of underlayer material, there was possibility of the occurrence of interfacial diffusion and other chemical reactions at the interface between NiAl and Ti. The objective of this work was to study the microstructure and properties of the synthesized products prepared using reactant pellets with different compaction pressures.

MATERIALS AND METHOD

Commercial carbon steel was used as substrate with diameter of 16 mm and thickness of 3 mm. The surface of substrate was grounded and polished to produce flat surface. Surface cleaning was accomplished in the acetone bath in ultrasonic cleaner for 15 minutes to remove dust, oil film, and grease from the surface. The moisture on the surface was then evaporated by drying in hot air. Nickel (Ni) and aluminium (Al) were used as the reactant for coating material, whereas titanium (Ti) was used as material for underlayer. After balanced with each specified weight based on the reaction equation of NiAl, the reactant powder was mixed with ceramic mortar. After the powder mixture has been thoroughly mixed, it was then balanced to produce 0.5 gram for each reactant sample. The powder was then cold compacted to form green pellet which carried out by using steel die package. The hydraulic pressure was used to press the pellet using compaction pressure of 100 MPa, 150 MPa and 200 MPa. The green density of underlayer after compacted using 100 MPa, 150 MPa and 200 MPa was 2125.71 ± 156.11 kg/m³, 2672.52 ± 285.57 kg/m³ and 2848.39 ± 213.79 kg/m³, respectively.

Figure-1 shows the schematic configuration of coating, underlayer, and substrate. Coating pellet is the mixture of Ni and Al with mole percentage of 1: 1, while the underlayer pellet is only composed by titanium



powder. Combustion process was carried out in a glove box with atmosphere from argon gas. An induction heating was used as ignition source to initiate the SHS process using a current of 300 A. Microstructure characterization was carried out by using optical microscope, scanning electron microscopy (SEM), and X-rays diffraction technique (XRD). The sample used for the microstructure characterization was cut in the cross sectional area of the synthesized product, and molded with epoxy resin, grounded with 180 and 600 grits of sand paper, and polished with a sequence of silica papers with diamond pasta down from 9, 3, to 1 micron. Etching of sample was done with methanol and nitrite acid with volume ratio of 98%: 2%. The measurement of the microhardness was conducted on the coating surface using a Vickers microhardness tester with a load of 0.098 N for 15 seconds.

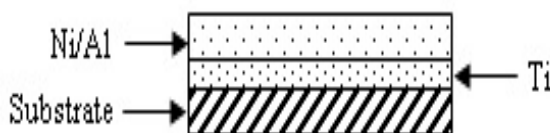


Figure-1. Schematic configuration of coating, underlayer, and substrate.

RESULTS AND DISCUSSION

Phase identifications

Preliminary observations on the SHS process of all samples showed that all compaction pressures have resulted in the completeness of the SHS process. This indicates that all compaction pressures have produced a sufficient density to cause the ignition and propagation of the SHS process. The completeness of the SHS reaction was indicated by the formed phase in the synthesized product as shown by XRD tests. Figure-2(a) and (b) show the XRD patterns obtained in the synthesized product at different depths from the surface. The XRD spectra of the product in Figure-2(a) show the formation of NiAl and $\text{Ni}_{0.58}\text{Al}_{0.42}$. This indicates that the SHS reaction of Ni/Al was complete producing NiAl and NiAl with Ni-rich. The formation of Ni-rich NiAl can be explained as the reduction of Al content due to a small quantity of Al has spread to the underlayer. The XRD spectra of the product in Figure-2(b) show the formation of Ti_2Ni , Ti_3Al , AlNi_2Ti which were formed as a reaction of Ni/Al in the upper layer and Ti in the underlayer, as previously reported (Riyadi, 2014). The appearance of Ti was attributed to the unreacted Ti underlayer. An unexpected $\text{Fe}_2\text{Ti}_4\text{O}$ was formed as oxides in the underlayer due to the reaction of Fe, oxygen and Ti underlayer.

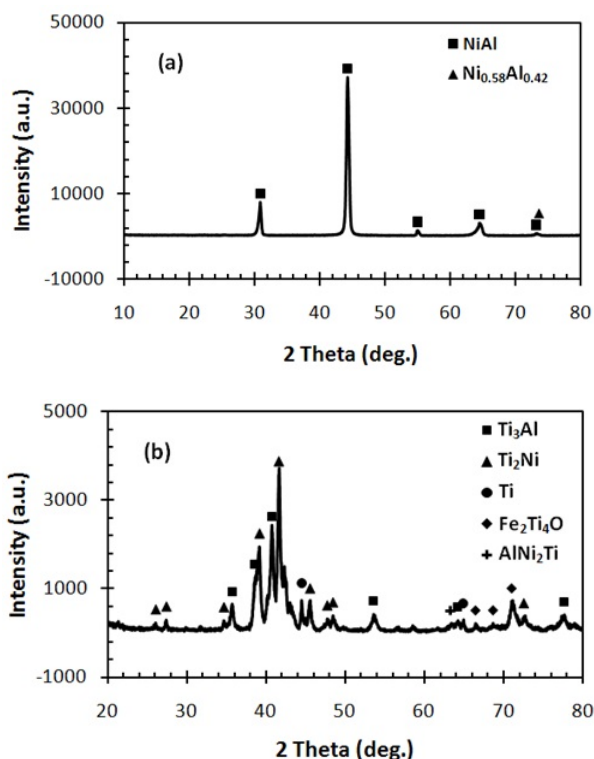


Figure-2. XRD spectra of the synthesized product observed at: (a) 40 μm ; and (b) 500 μm from the surface.

Microstructure distributions

The typical microstructure of the synthesized products obtained by compaction pressure of 100 MPa, 150 MPa and 200 MPa were observed using SEM images as shown in Figure-3(a), (b) and (c). The result shows that the variation of compaction pressure has significantly affected the microstructure of synthesized product, particularly, on the amount of Ti alloy and the product porosity. The result in this study shows that the different amount of Ti alloy can be observed in all products. The appearance of Ti alloy in all samples indicates that the heat produced by the Ni/Al reaction was insufficient to promote further reactions between the liquid Ni/Al and all amount of titanium underlayer. However, a comparison of the result shows that the amount of Ti alloy in the compaction pressure of 150 MPa is higher than that in the samples compacted by 100 and 200 MPa. In other words, an increase of compaction pressure initially increased the amount of Ti alloy, and further increase of the compaction pressure resulted in a reversed trend by producing a smaller amount of the Ti alloy. This trend may occur as a result of the thermal conductivity of the samples which generated two opposing effects simultaneously between the amount of the heat transferred to the underlayer and the heat lost to the environment. The heat released by Ni/Al reaction which transferred into underlayer was dependent on the thermal conductivity of the compact. An increase in the compaction pressure led to an improvement in the particle contacts which hence increased the thermal



conductivity (Adeli *et al.* 2010). A high amount of Ti alloy in the sample with a compaction pressure of 150 MPa may be due to the amount of heat lost to the surrounding was higher than the heat conducted to the underlayer, which in turn, became a barrier to the reaction of the liquid Ni/Al and Ti. Further increase in the compaction pressure (200 MPa) resulted in an increase in the thermal conductivity which was favoured to the heat transfer and consequently promoted the reaction between the liquid NiAl and Ti. The result in this study also shows that an increase in the compaction pressure led to an increase in the formation of pores in the synthesized product. As mentioned in some literatures, there are a number of sources leading to the formation of porosity (Tay *et al.* 2008)(Gao *et al.* 2013). In SHS product, the porosity is generally formed due to the existing pores in the compacted reactant. An increase in the compaction pressure should therefore decrease the porosity since it decreases the distance between particles resulting in the reduction of the inter-particle pores. The present work, however, produces a contradictive result. The result indicates that the porosity of synthesized product increases with an increase in the compaction pressure (see Figure-3(c)). This may be due to an increase of the compaction pressure produced a high thermal conductivity which resulted in a high heating rate and a short time of the reaction. Due to insufficient time, the formed pores could not rise to the surface and resulted in the formation of porous product.

Microhardness

Figure-4 shows the microhardness of synthesized products that was measured on the coating surface. The result shows that an increase in the compaction pressure of the reactant pellet produced a fluctuation of the coating hardness. The microhardness of the coating decreases from 350 HV to 302 HV when the reactant pellet was compressed by 100 MPa and 150 MPa. However, when the pellet was compressed using 200 MPa, the hardness of coating increases to 348 HV. This fluctuation can be correlated to the content of the formed phase of synthesized product below the coating. This phenomena needs further study related to the effect of the hardness of phases below the measured one.

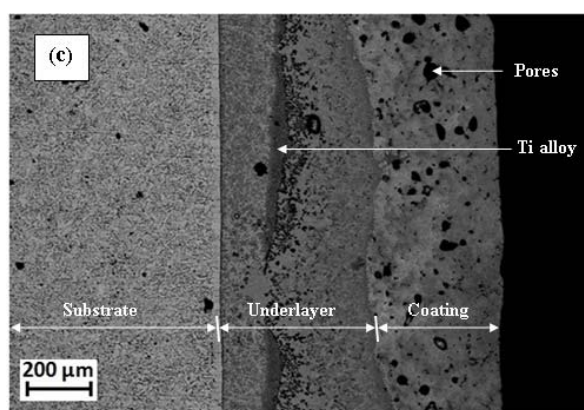
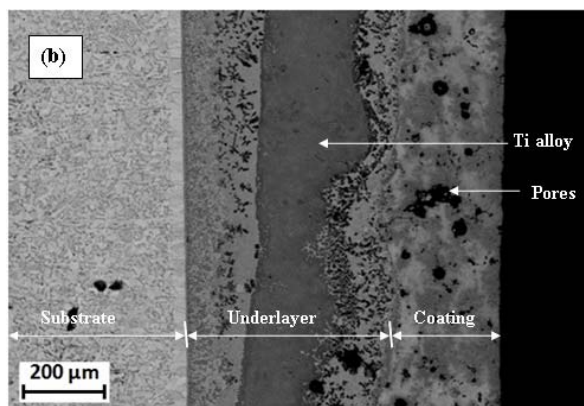
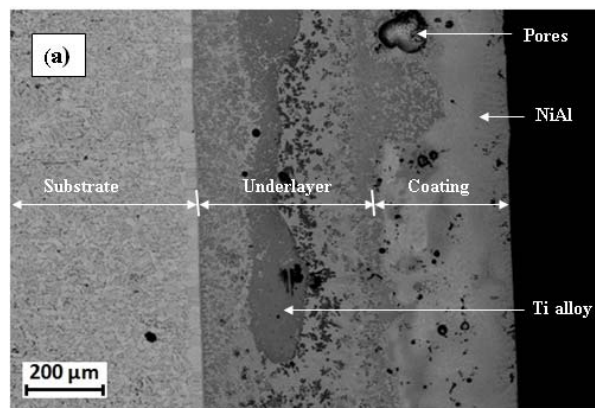


Figure-3. Back scattered SEM micrograph of synthesized products prepared by compaction pressure of: (a) 100 MPa; (b) 150 MPa, and (c) 200 MPa.

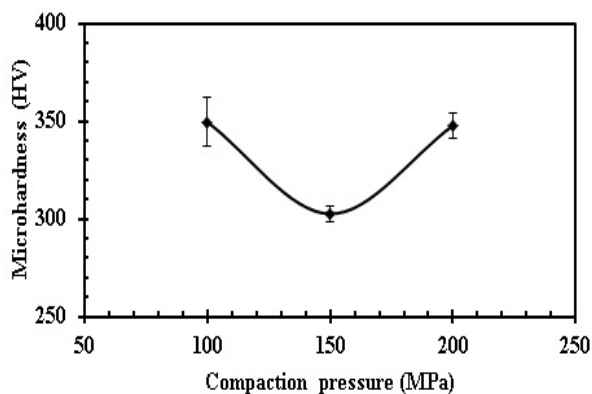


Figure-4. Microhardness of NiAl produced by varied compaction pressures.

CONCLUSIONS

The effect of compaction pressure on the microstructure and properties of the product formed by SHS process was successfully observed. The reactants that were composed of Ni/Al and Ti layers have reacted producing several intermetallic materials. The compaction pressure significantly affected the distribution of synthesized products which related to the quantity of Ti



alloy and porosity. The porosity increased with an increase of the compaction pressure of the reactant pellets. The hardness of the coating fluctuated with the increase of the compaction pressure. Considering that the compaction pressure of the reactant for SHS process significantly affected the microstructure and properties of the synthesized product, an optimized compaction pressure of the reactant pellet can then be used to improve the performance of the product.

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