Proceedings of the 2nd International Congress APMAS2012, April 26–29, 2012, Antalya, Turkey

Characterization of Ni_3Al and Ti_3Al Coatings Produced by Electric Current Activated Sintering Method

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The present study reports on Ni_3Al and Ti_3Al-based intermetallics coated on AISI 1010 steel substrate by one-step pressure assisted electric current activated sintering method. Ni, Ti, and Al elemental powders were mixed in the stoichiometric ratio corresponding to the Ni_3Al and Ti_3Al intermetallics at molar proportion of 3:1. The mixed coating powders were placed onto the steel substrate in a mold, and pressed with compressive stress of 100 MPa and then, electric current: 1100–1200 A, voltage 2.9–3.4 V was applied for 15 min during coating. As-synthesized coatings seem to have good adherence with many small porosities. The phases formed in the Ni_3Al coating layer were Ni_3Al as a major phase beside NiO and $NiAl_2O_4$ trace phases, which were confirmed by X-ray diffraction analysis. However, TiAl and Ti trace phases besides major Ti_3Al phase were detected in the Ti_3Al coating. The hardness of the Ni_3Al, Ti_3Al coatings and AISI 1010 substrate was approximately 321 ± 18, 445 ± 13 and 157 $HV_{0.5}$, respectively.

DOI: 10.12693/APhysPolA.123.245

PACS: 81.20.Ev, 81.40.Pq

1. Introduction

Intermetallic materials, especially transition-metal (Fe, Ni, Ti, and Co) aluminides, continually attract attention of material's scientists because of a potentially very attractive set of physical, chemical, thermal and mechanical properties. Among the monolithic intermetallic materials, aluminides are important for industrial applications due to their low cost, relatively high melting point, good wear resistance, ease of fabrication, and high-strength to weight ratio. In addition, they exhibit excellent corrosion resistance in oxidizing and sulfidizing atmospheres owing to the formation of protective Al_2O_3 scale. Its use as a coating material is justified in applications where wear, high-temperature oxidation, and electrochemical corrosion may be present [1–9].

Currently, aluminides coating can be manufactured by using several techniques such as PVD, reactive sintering, etc. Among these, electric current activated/assisted sintering (ECAS) technique, loose powders or a cold formed compact to be consolidated are inserted into a container which is heated to and then held at the desired temperature, while pressure is applied and maintained for a given period of time. Heat is provided by passing an electric current through the powders and/or their container, thus exploiting the consequent Joule effect. It simultaneously applies an electric current along with a mechanical pressure in order to consolidate powders or synthesize and simultaneously densify specific products with desired configuration and density [10–12].

The main aim of the present study is to characterize Ni_3Al and Ti_3Al coating produced on AISI 1010 steel substrate from Ni, Ti, and Al precursors by one-step pressure assisted electric current-assisted sintering.

2. Experimental details

Carbonyl–Ni powders (99.8% purity, 4–7 μm), Ti (99.5% purity, less than 44 μm) and gas-atomized aluminum powder (99% purity and 15 μm grain size) were used as starting powders mixtures to produce Ni_3Al- and Ti_3Al-based intermetallic coatings. The started powders were mixed in stoichiometric ratio corresponding to the Ni_3Al and Ti_3Al intermetallic phases, in a molar proportion of 3:1. In order to obtain uniform mixtures, the powder mixtures were ball-milled in the Ar+2%H_2 gas atmosphere with some amounts of ethanol addition for the period of 10 min. Commercial carbon steel AISI 1010 was used as the substrate, which was machined as a disk 10 mm diameter and 5 mm in thickness. Before the coating process, all substrates were ground using 600 grid emery paper to obtain a good surface finish. AISI 1010 carbon steel was placed on a punch in the mold with the diameter of a 15 mm which was lubricated with a thin layer of boron nitride. The mixed coating powders were placed onto the substrate specimen in the mold. The specimen was kept in an open atmosphere, and then in order to provide the contacting of individual powders in the initial period of the process the sample was pressed by a die with a compression load of 100 MPa, for 1 min. A direct electric current (1100–1200 A, voltage 2.9–3.4 V) was applied to the steel substrate with a pressure of 25 MPa for 15 min in order to manufacture coatings. After sintering, the specimens were unloaded and cooled to room temperature in an open atmosphere. The morphologies of the samples and the presence of phases formed were examined by using scanning electron microscopy (SEM), optical microscopy (OM) and X-ray diffraction (XRD)

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analysis. Microhardnesses of the test materials were measured using a Vickers indentation technique with a load of 0.5 N.

3. Results and discussion

Pressure-assisted electric current-activated sintering process is combined effects of both the electric current and pressure. The electric current was used for supplying the energy needed to complete the reaction and to generate the Ni_3Al- or Ti_3Al-based coating *in situ*. In order to decrease the amount of porosity and obtain a dense coating structure a pressure is directly applied on the powders placed on steel, which are in the mould [13]. The morphology of the interface between the coating and the substrate were examined by using SEM and OM in Fig. 1a,b. The microstructures of Ni_3Al- and Ti_3Al-based intermetallics coatings formed on the surface of the substrate are given in Fig. 1. As it can be seen in Fig. 1, the Ni_3Al intermetallics coating layer consist of many pores. The Ti_3Al intermetallics coating layer including small amount of pores possessed a coarser grain structure than the Ni_3Al intermetallics coating layer (Fig. 1b).

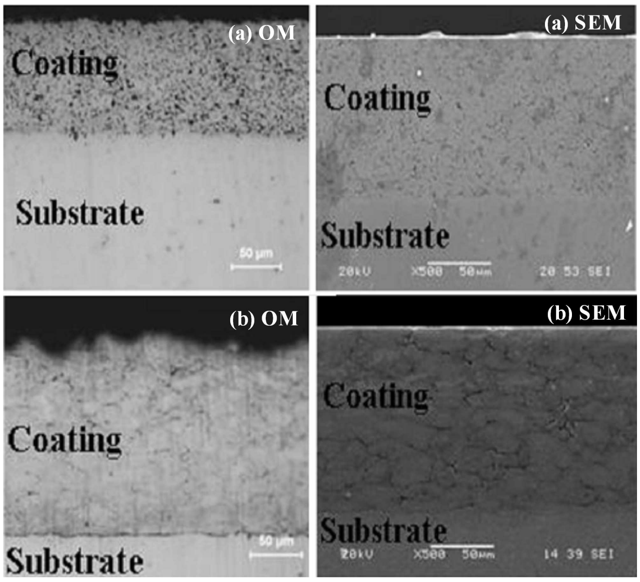

Fig. 1. OM and SEM images of coated samples: (a) Ni_3Al and (b) Ti_3Al.

As it can be seen in Fig. 1, there are no pores and cracks at the interface between the matrix and coating layer, which shows that the interface bonding is good. In addition, the Vickers indenter was applied on the interface of steel matrix and coating layer under the loads of 10 N and any cracks was not formed at interface as shown in Fig. 2. So, the samples seem to have good adherence at the interface between the matrix and coating layer.

The porosities in coatings originated from pores initially present in the green compact and from outgassing

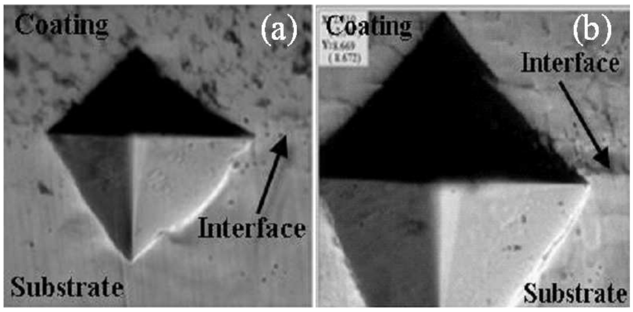

Fig. 2. Vickers indentations marks applied on the interface of coating layers of (a) Ni_3Al and (b) Ti_3Al.

or the vaporization of impurities at the high temperatures reached during the reaction. Figure 3a,b illustrates the XRD pattern of the coatings.

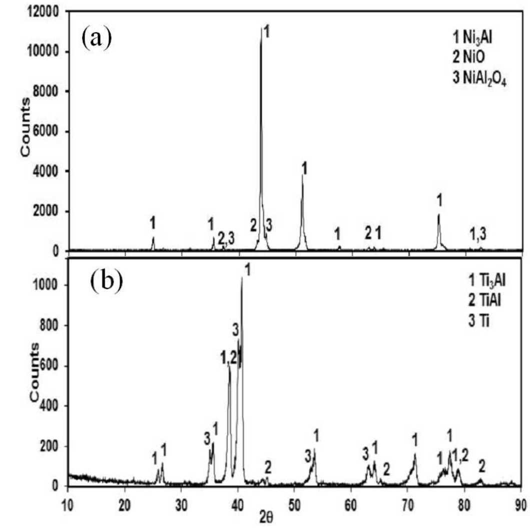

Fig. 3. X-ray diffraction patterns of coated samples: (a) Ni_3Al and (b) Ti_3Al.

It can be seen that the Ni_3Al-based coating consists of Ni_3Al as a major phase, along with NiO and $NiAl_2O_4$ trace phases (Fig. 3a). TiAl and Ti trace phases were detected along with the major Ti_3Al phase in the Ti_3Al-based coating (Fig. 3b). The process is very fast and the densification of coating layer was realized in 15 min. But, the reaction was not completed in the Ti_3Al coating process. It is possible to claim that the holding time or applied current is insufficient for the formation of Ti_3Al phases in the coating process, completely. Coating process was carried out in an open atmosphere without protective gas. Probably, this should be reason for Ni oxides realized in the process which was identified by XRD analysis technique.

The hardness of the Ni_3Al-, Ti_3Al-based coatings and AISI 1010 substrate was approximately 321 ± 18, 445 ± 13 and 157 $HV_{0.5}$, respectively.

4. Conclusions

The following results can be derived from the present study.

Ni_3Al and Ti_3Al coatings on the AISI 1010 carbon steel produced by the one-step pressure-assisted ECAS method, successfully.

The microstructures of the coatings have a low porosity and seem to have good adherence at interface.

The phases formed in the Ni_3Al coating layer included Ni_3Al as a major phase, along with NiO and $NiAl_2O_4$ trace phases. For the Ti_3Al coating, TiAl and Ti trace phases along with a major Ti_3Al phase were detected. These findings were confirmed by XRD analysis.

Acknowledgments

This work was supported by the Sakarya University Scientific Research Foundation (project number: 2008--50-01-013).

References

[1] N. Stoloff, C.T. Liu, S.C. Deevi, *Intermetallics*, Elsevier Sci., 2000, p. 1313.

[2] V. Paidar, *Mater. Sci. Eng. A* **234-236**, 15 (1997).

[3] K. Vedula, in: *Intermetallic Compounds, Structural Applications of Intermetallic Compounds*, Eds. J.H. Westbrook, R.L. Fleischer, 1995, p. 203 .

[4] G. Sauthoff, *Intermetallics* **8**, 1101 (2000).

[5] W. Godlewska, S. Szczepanik, R. Mania, J. Krawiarz, S. Kozinski, *Intermetallics* **11**, 307 (2003).

[6] J.Y. Rhee, *Curr. Appl. Phys.* **6**, 852 (2006).

[7] Y.B. Pithawalla, M.S. El Shall, S.C. Deevi, *Intermetallics* **8**, 1225 (2000).

[8] A.C. Lilly, S.C. Deevi, Z.P. Gibbs, *Mater. Sci. Eng. A* **258**, 42 (1998).

[9] W.J. Zhang, R.S. Sundar, S.C. Deevi, *Intermetallics* **12**, 893 (2004).

[10] N. Ergin, O. Ozdemir, C. Bindal, in: *5th Int. Powder Metallurgy Conf., Turkey*, Ankara 2008, p. 917.

[11] R. Orru, R. Licheri, A.M. Locci, A.G. Cincotti, *Mater. Sci. Eng. R* **63**, 127 (2009).

[12] J. Rodriguez, S.O. Moussa, J. Wall, K. Morsi, *Scr. Mater.* **48**, 707 (2003).

[13] R. Orru, R. Licheri, A.M. Loci, A. Cincotti, G. Cao, *Mater. Sci. Eng. R* **63**, 127 (2009).