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EFFECT OF ANNEALING ON THE STRESS–STRAIN RESPONSES AND FRACTURE OF TI/CU/TI CLAD COMPOSITE

Yong Keun Kim and Sun Ig Hong*

Abstract: *The effect of annealing on the stress-strain responses and fracture of Ti/Cu/Ti clad composite was investigated. The intermetallic layer thickness increased with increase of annealing temperature above 700°C. The increase of hardness at 700 °C indicates the appreciable growth of intermetallic compounds at the interface (8ìm). Four intermetallic layers, Cu_4Ti, Cu_3Ti_2, CuTi and $CuTi_2$ were suggested to be present at the Cu/Ti interface based on the XRD. No emanating cracks from the corners of the indentation mark, which is typically observed in the clad with brittle intermetallic layers suggests the intrinsic non-brittle nature of intermetallics at the Ti/Cu interface. Stress-strain response of as-rolled Ti/Cu/Ti clad composite exhibited the brief work hardening up to 550MPa accompanied by rapid work softening, resulting in low ductility (~5%). With post-cold-rolling heat treatment, the stress-strain curves exhibited the extended work hardening. The strength decreased to 253MPa and the ductility increased to ~43% after annealing at 700°C. No stress drops were observed in the stress-strain curves until the final fracture, indicating that Cu and Ti fracture simultaneously at the final moment. The excellent interface bonding strength and the formation of non-brittle intermetallics in Ti/Cu/Ti ensures the mechanical reliability in Ti/Cu/Ti clad composite.*

Keywords: *Titanium, Copper, Heat-treatment, Clad, Composite, fracture*

INTRODUCTION

The demand for multifunctional clad composites with enhanced properties and various functions has increased driven by the needs in various industries (Kim W.N. & Hong S.I.: 2016, Ha J. S. & Hong S.I. : 2016, Jin J.Y. & Hong S. I. : 2014, Manesh H.D. & Taheri A.K. : 2003,

Lee K.S. & Lee S.E. & Sung H.K. & Lee D.H. & Kim J.S. & Chang Y.W. & Lee S. & Kwon Y.N. : 2013, Peter C. Tortorici & M.A. Dayananda. : 1998). The clad metals consisting of two or more metal layers have been developed because of their unique combination of properties such as high strength, high conductivity and excellent corrosion resistance (Kim I. K. & Hong S.I. : 2014, Lee K.S. & Yoon D.H. & Kim H.K. & Kwon Y.N. : 2012, Kim I. K. & Hong S.I. : 2013, Kim I. K. & Hong S.I. : 2013, Kim H. B. & Hong S.I. : 2015). The properties of clad composites are determined by

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the selection of component materials to be joined and the stacking structure of different materials with various thicknesses and the interface structure and the properties between different materials (Kim I. K. & Hong S.I. : 2014, Lee K.S. & Yoon D.H. & Kim H.K. & Kwon Y.N. : 2012, Kim I. K. & Hong S.I. : 2013). Clad composites with different metals and alloys along with various combinations of properties have been commercialized and used in various industrial fields (Kim I. K. & Hong S.I. : 2014, Lee K.S. & Yoon D.H. & Kim H.K. & Kwon Y.N. : 2012, Kim I. K. & Hong S.I. : 2013, Kim I. K. & Hong S.I. : 2013, Kim H. B. & Hong S.I. : 2015; Hayeeye, Yu, Sattar, Chinpa & Sirichote, 2015).

Titanium has high strength and corrosion resistance and copper has excellent electrical conductivity. Ti/Cu clad composite has been used effectively as an electrode material for chemical processing in corrosive environments such as chlor-alkali, electrolysis, metal recovery, and plating facilities. Titanium cladding can be used to increase both the mechanical strength and the corrosion resistance of the clad composite (F. Xu & J. C. Fredette & R. A. Holt & R. B. Rogge & D. Pickard & L. Tuck : 2007) The aim of this study was to investigate the effects of annealing on stress-strain responses and microstructure of 3-ply Ti/Cu/Ti clad metal fabricated by roll-bonding.

EXPERIMENTAL METHOD

The material used in this work was 3-ply Ti/Cu/Ti layered clad composite produced by warm-roll-bonded and cold-rolled with a reduction ratio of 70%. Clad plate had a total thickness of about 1mm and the thickness of Ti and Cu plate were 0.1mm and 0.8mm, respectively. Ti/Cu/Ti plate was heat-treated at various temperatures (300, 500, 700 °C) for 2 hour to observe the effect of heat-treatment. To investigate the mechanical properties of annealed clad composites at various temperatures, tensile tests were performed using a Universal Materials Testing Machine (UNITED) at room temperature. The gage length and gage width for the tensile testing sample were 14.7mm and 3.4mm, respectively. During the test, the strain rate was $1x10^{-3/s}$. Hardness testing of heat-treated Ti/Cu/Ti clad composites was performed using Vickers hardness tester. To analyze the interfacial microstructure after heat treatment and cross-sectional fracture surface of clad material, Optical Microscope and FE-SEM were used.

RESULT AND DISCUSSION

In Fig. 1, stress–strain curves of the as-rolled clad plates and those annealed at various temperatures are exhibited. A step-wise drop in the stress-strain curves observed in Mg/Al/STS clad composite (Kim I. K. & Hong S.I. : 2013) did not occur in the Ti/Cu/Ti clad composite, suggesting the simultaneous fracture of Ti and Cu due to the excellent interface bonding. Tensile strength and ductility of cold-rolled specimen was observed to be 562MPa and 5%, respectively. Ti/Cu/Ti

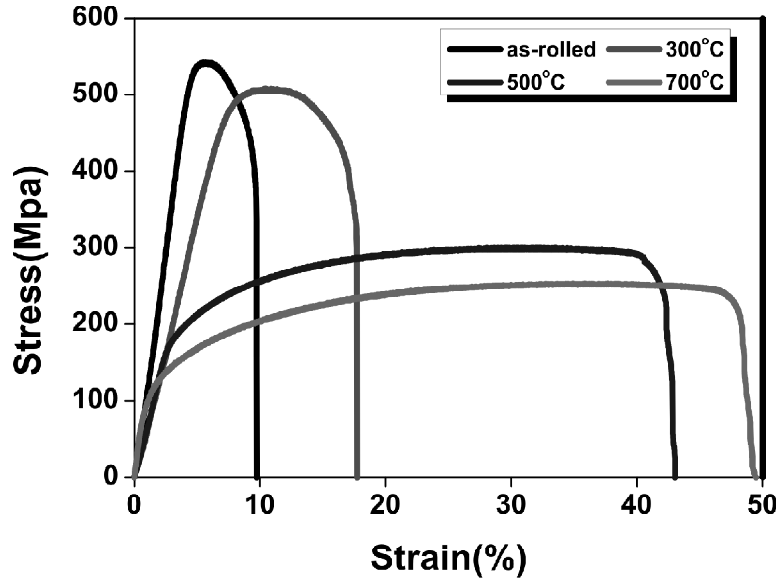

Figure 1: Stress–strain curves of as-rolled and annealed Ti/Cu/Ti composites at 300 °C, 500°C, and 700°C

clad composite after heat treatment at 700 °C exhibited the tensile strength of 253MPa and 43% ductility. As expected, the strength decreased and the ductility increased with increase of heat treatment temperature. It is interesting to note that the reduction of ductility observed after high temperature annealing in other clad composites (Kim I. K. & Hong S.I. : 2013, Kim I. K. & Hong S.I. : 2014) was not observed in Ti/Cu/Ti clad composite of the present study. The ductility continued to increase even after annealing at 700 °C.

Fig. 2 shows the variation of Vickers micro hardness of Ti layer, Cu layer and the Ti/Cu interface as a function of heat treatment temperature. The drops of hardness value in Ti and Cu at 500 °C are attributed to the recrystallization of Ti and Cu. The hardness of interface region also decreased with the decrease of hardness in Ti and Cu layers, reflecting the overall softening in Cu and Ti matrix. One interesting observation in Fig. 2, however, is the increase of hardness at the Ti/Cu interface of Ti/Cu/Ti clad composite annealed at 700!. The increase of hardness at 700! indicates the growth of intermetallic compounds at the interface. The highest hardness value (~126 Hv) at the interface after heat treatment at 700!, although it is greater than that at the interface of as-cold-rolled clad composite, is still lower than the hardness values (~220Hv) of Ti matrix before recrystallization. The highest hardness value at the interface in Fig. 2 is far smaller than that (~400 Hv) of the interface observed in Cu/Al/Cu clad composite (Kim I. K. & Hong S.I. : 2014, Kim I. K. & Hong S.I. : 2012). The smaller hardness at the interface of Ti/Cu/Ti clad composite suggests that the intermetallics is not as brittle as those

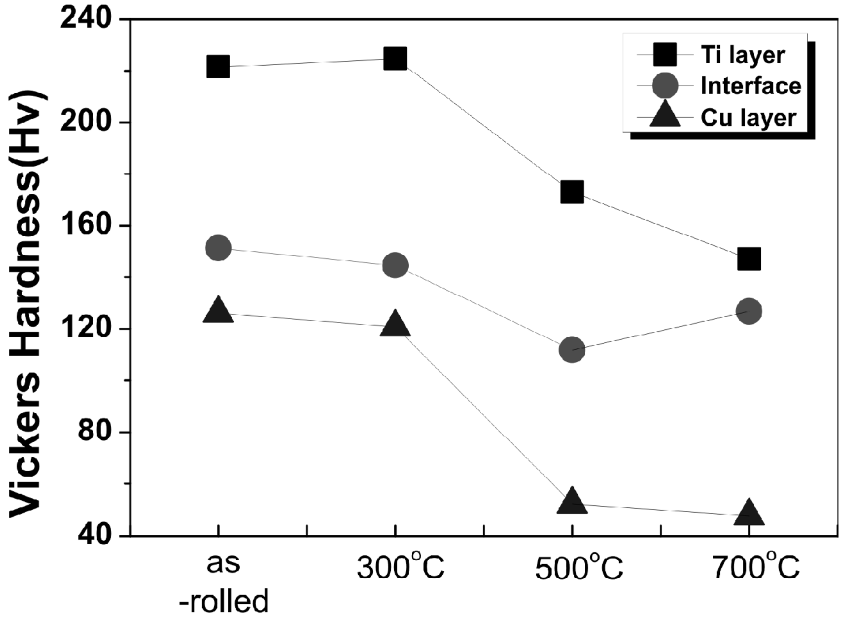

Figure 2: Vickers micro hardness of Ti/Cu/Ti clad composite as a function of heat treatment temperature

observed in Cu/A/Cu(Kim I. K. & Hong S.I. : 2014, Kim I. K. & Hong S.I. : 2013, F. Xu & J. C. Fredette & R. A. Holt & R. B. Rogge & D. Pickard & L. Tuck : 2007, Kim I. K. & Hong S.I. : 2012) and Mg/Al/STS(Kim I. K. & Hong S.I. : 2014, Kim I. K. & Hong S.I. : 2013).

Fig. 3 shows the Vickers micro hardness indentation mark at the interface of Ti/Cu/Ti clad composite; heat treated at 500 °C and 700 °C. No interfacial cracks were induced by the indentation in Ti/Cu/Ti clad composite heat-treated at 500! and at 700 °C Ti/Cu/Ti clad composites, indicating the sound interfacial bonding. In the heat-treated Ti/Cu/Ti clad composite at 700 °C with the appreciable intermetallic layer formation, no emanating cracks from the corners of the indentation mark, which is typically observed in the clad with brittle intermetallic layers, were observed, suggesting the intrinsic non-brittle nature of intermetallics at the Ti/Cu interface. The absence of cracks from the corners of the indentation mark in Fig. 3 indicates that intermetallics in Ti/Cu/Ti clad are ductile enough to accommodate the micro-plastic flow from indentation.

Fig. 4(a)~4(d) display the FESEM images of the near-interface region of Ti/Cu/Ti clad composite; as-cold-rolled (a) and heat-treated at 300 °C (b), 500 °C (c) and 700! (d). Fig. 4(e)~4(h) show the near-fracture region of fractured Ti/Cu/Ti clad composite after tensile testing; as-cold-rolled (e) and heat-treated at 300 °C (f), 500 °C (g) and 700! (h). The FESEM images in Fig. 4 (a) ~ (d) clearly revealed

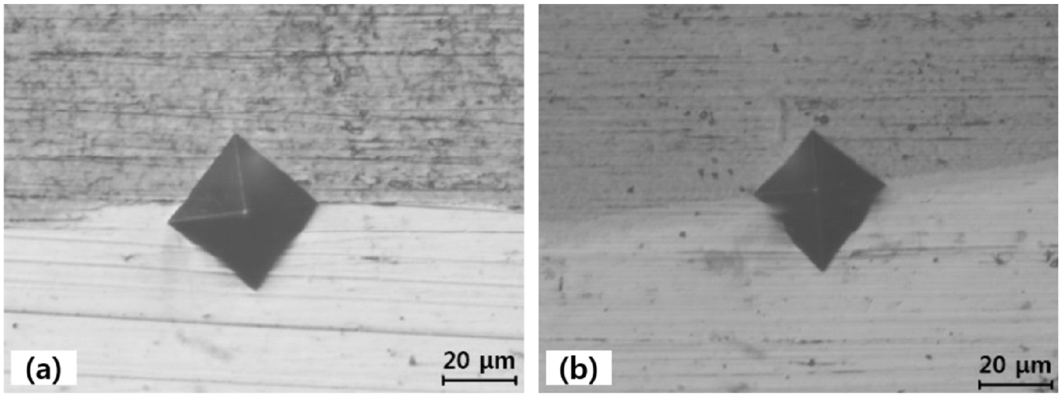

Figure 3: Vickers micro hardness indentation mark at the interface of Ti/Cu/Ti clad composite; heat treated at 500 °C (a) and 700 °C(b)

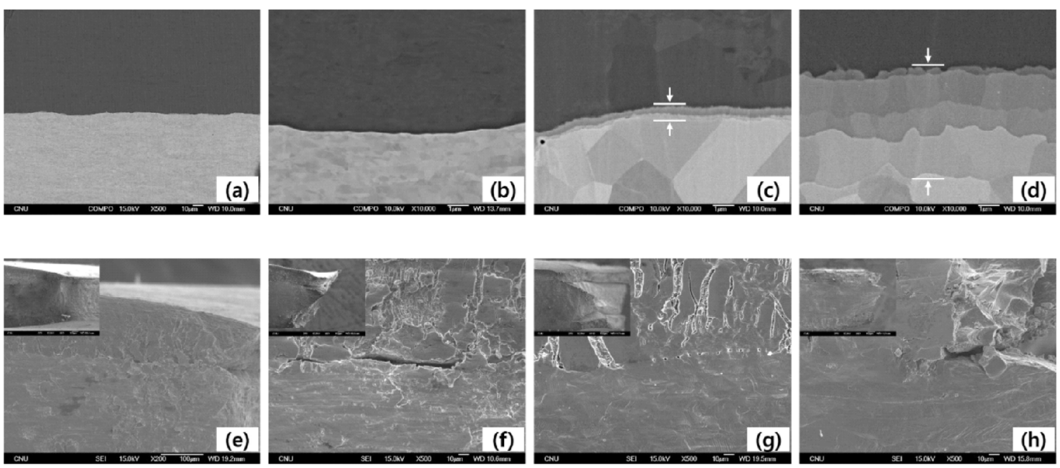

Figure 4: FESEM images of the near-interface region of Ti/Cu/Ti clad composite; as-cold-rolled (a) and heat treated at 300°C (b), 500°C (c) and 700°C (d) and near-fracture region of fractured Ti/Cu/Ti clad composite after tensile test; as-cold-rolled (e) and heat treated at 300°C (f), 500°C (g) and 700°C (h)

that there is no visible intermetallic compound formation up to 300!. Above 500!, a very thin intermetallic compound layer was observed. The intermetallic layer grew very rapidly after 700 °C and its thickness reached ~8¼m after heat treatment. It should be noted that the grain size in Cu layer increased rapidly with increase of annealing temperature. Coarse slip traces were observed in Cu layer after annealing above 500 °C. The presence of slip traces after annealing at high temperatures is attributed to the rather easy slip (Hong S.I. & C Laird : 1990) in the coarse-grained Cu.

Despite the thickness-wise neck formation in the as-rolled plate(e) and those annealed at 300 °C (f), 500 °C (g) and 700 °C (h) no pronounced interface debonding and separation between Ti and Cu layers were noted, supporting an reliable interface bonding between Ti and Cu. Ti and Cu layers in the as-rolled Ti/Cu/Ti composite. It is obvious that Ti layers and Cu layers co-deformed and co-fractured as if they are a single material. The observation of no interface debonding and separation is supported by the presence of no appreciable interfacial intermetallic layers after annealing at and below 500°C. Fig. 4(e) shows no strain incompatibilities and cracks were observed across the interface in the as rolled clad composite.

In order to identify the thin intermetallic compounds more accurately if there are any, bi-layered Ti/Cu composite plates were separated along the Ti/Cu interface or through the ductile intermetallic layers between Ti/Cu. Fig. 5(a) and 5(b) show the XRD patterns from the surfaces of peeled-off Ti plate (a) and Cu plate(b) of Ti/Cu/Ti composite heat-treated at 700 °C for 2hours. The fractured interfaces of two separated layers, Ti side and Cu side with exposed intermetallics on the surface were examined by XRD analysis because the intermetallic layer is very thin. Fig. 5(a) and 5(b) demonstrate the presence of $CuTi_2$ and Cu_3Ti_2 in addition to Ti on the Ti side and Cu_4Ti and Cu_3Ti_2 (Okamoto H. : 2002) in addition to Cu on the Cu side of Ti/Cu/Ti composite heat-treated at 700 °C. The presence of Cu_3Ti_2 peak detected on both Ti and Cu side of peeled-off plates suggests that the fracture did not take place in Ti and Cu plates and cracks propagated in the intermetallic layers. The presence of Cu_3Ti_2 on both peeled-off Ti and Cu plates indicates that the most brittle phase could be Cu_3Ti_2.

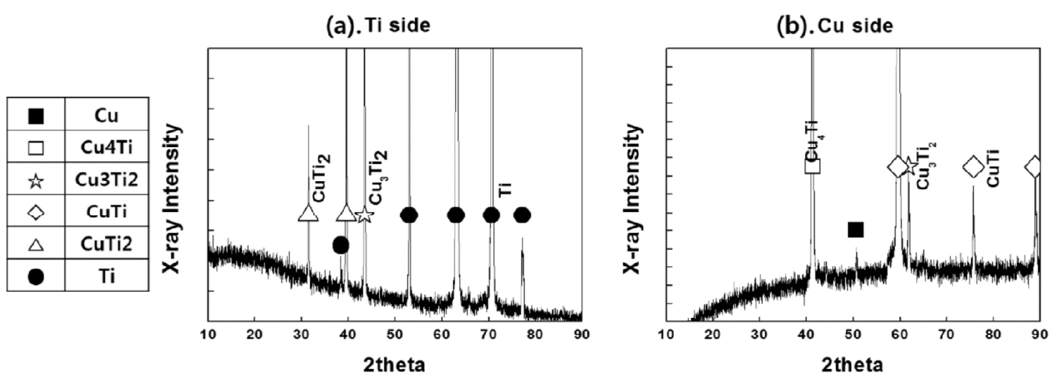

Figure 5: XRD patterns from the interface region of separated Ti (a) and separated Cu plate (b) from heat-treated at 700°C for 2h.

SUMMARY AND DISCUSSION

As a result of the study on the tensile properties and microstructure of Ti/Cu/Ti clad composite, the following conclusions are obtained:

[1] Tensile strength and ductility of cold-rolled specimen was observed to be 562MPa and 5%, respectively. Ti/Cu/Ti clad composite after heat treatment at 700! exhibited the tensile strength of 253MPa and 43% ductility. The ductility continued to increase even after annealing at 700^{o}C.

[2] The highest hardness value at the interfacial intermetallic layer (~126 Hv) is far smaller than that (~400 Hv) of the interface observed in Cu/Al/Cu clad composite. The smaller hardness at the interface of Ti/Cu/Ti clad composite suggests that the intermetallics is not as brittle as those observed in Cu/Al/Cu and Mg/Al/STS.

[3] No emanating cracks from the corners of the indentation mark, which is typically observed in the clad with brittle intermetallic layers suggests the intrinsic non-brittle nature of intermetallics at the Ti/Cu interface. The absence of cracks from the corners of the indentation mark indicates that intermetallics in Ti/Cu/Ti clad are ductile enough to accommodate the micro-plastic flow from indentation.

[4] The presence of $CuTi_2$ and Cu_3Ti_2 were confirmed on the peeled-off Ti plate of Ti/Cu/Ti clad composite whereas the presence of Cu_4Ti and Cu_3Ti_2 were confirmed on the peeled-off Cu plate of Ti/Cu/Ti composite heat-treated at 700!. Four intermetallic layers, Cu_4Ti, Cu_3Ti_2, CuTi and $CuTi_2$ were suggested to be present at the Cu/Ti interface based on the XRD

[5] The presence of Cu_3Ti_2 peak was detected on both Ti and Cu side of peeled-off plates suggests that the fracture did not take place in Ti and Cu plates and cracks propagated in the intermetallic layers. The presence of Cu_3Ti_2 on both peeled-off Ti and Cu plates indicates that the most brittle phase could be Cu_3Ti_2.

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