Deformation behavior of an electrodeposited nano-Ni/amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ laminated composite sheet

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Abstract – A nano-Ni/amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ composite sheet was prepared in the form of three-ply (Ni- $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ -Ni) laminated structure by an electrodeposition method. The average grain size of Ni layers is about 50 nm. The interface of laminated composite was investigated with SEM equipped with energy dispersive scanning (EDS) and line analysis technique. The laminated composite has a good interfacial bonding between amorphous layer and nano-Ni layers due to the mutual diffusion of atoms in $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ and Ni layers during the process of electrodeposition. A maximum elongation of 115.5% was obtained when the volume fraction of nano-Ni layers (V_{Ni}) was 0.77, which is greatly higher than that of monolithic amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ ribbon (36.3%) tested under the same conditions. Bulging tests were carried out to evaluate plastic forming properties of the $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ /Ni laminated composite. Under the condition of 450 °C, 4.0 MPa and 30 min, a good bulging part with the relative bulging height (RBH) of 0.4 was obtained.

Key words: Electrodeposition, Nano-material, Amorphous, Laminated composite

1. Introduction

The strength, hardness, shock crack energy and corrosion resistant property of amorphous alloys, compared to corresponding crystalline alloys, have obvious advantages [1–5]. However, the ductility of amorphous alloys is very limited due to the highly localized plastic flow, which occurs by the nucleation and propagation of shear bands [6–8]. Therefore, brittleness is regarded as an intrinsic problem of amorphous alloys. Amorphous/metal laminated composites (AMLCs) have been investigated in order to improve the ductility and fracture toughness of amorphous alloys at room temperature. Some researchers have produced AMLCs by a few novel methods for improving the tensile ductility of amorphous alloys. For example, Alpas and Embury [9] produced laminated composite structures of amorphous $\text{Ni}_{78}\text{Si}_{10}\text{B}_{12}$ -Cu by electrodeposition and diffusion bonding. Leng and Courtney [10, 11] prepared brass (Cu-30%Zn)-nickel base metallic glass ($\text{Ni}_{91}\text{Si}_7\text{B}_2$) composite laminates by soldering these constituents together with a Pb-Sn alloy. Nieh [12, 13] synthesized nanocrystalline Cu/amorphous Cu-Zr laminate by magnetron sputter deposition and found the laminate possesses a very high yield and tensile strength while still retaining a reasonable tensile elongation (4%).

In the present study, in order to improve the tensile ductility of amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ ribbon, nano-Ni layers were electrodeposited on the amorphous alloy ribbon to produce amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ /nano-Ni laminated composite. The effects of temperature, initial strain rate and V_{Ni} on tensile properties of the laminated composite at high temperature were investigated. The mechanism of the tensile conformity also was discussed. The bulging tests were also carried out to evaluate the plastic forming properties.

2. Experiment

The substrate for electrodeposited Ni layers was $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ ribbons prepared by melt spinning. The samples of the ribbons were 20 mm wide and 30 μm thick. X-ray diffraction confirmed that they were amorphous in the as-received state. The crystallization temperature (T_x) of amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy is about 540 °C, as determined by DSC analysis with a heating rate of 20 °C/min. Ni was electrodeposited onto amorphous ribbon substrates by pulse current electrodeposition in a plating bath. The electrodeposition bath composition was 300 g/L nickel sulfamate, 15 g/L nickel chloride, 30 g/L boric acid, 1 g/L saccharin, and 0.5 g/L sodium dodecyl sulfate. The bath temperature was kept at 50 ± 1 °C and magnetically stirred at 600 rpm. A commercial Ni plate with a purity of 99.6% was used as the anode and $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ ribbon as the

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Figure 1. Schematic diagram of the gas pressure bulging die.

Figure 2. The microstructure of amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ /nano-Ni laminated composite: (a) cross-sectional view and (b) TEM morphology of electrodeposited Ni layer corresponding to circle region in (a).

Figure 3. Tensile specimens of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ /Ni laminated composite shown in the as-machined geometry and after tension deformed at a strain rate of $8.3 \times 10^{-4} \text{ s}^{-1}$.

Figure 4. True stress-strain curves under different temperatures, at the strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$.

cathode. The samples of the ribbon were 20 mm wide and 30 μm thick. X-ray diffraction confirmed that the ribbons were amorphous in the as-received state. The amorphous ribbon, due to its thin thickness, was adhered to an Al plate. Electrodeposition was carried out using square-wave pulse current with 50% duty cycle (pulse on-time $T_{\text{on}} = 100 \text{ ms}$, pulse off-time $T_{\text{off}} = 100 \text{ ms}$) and constant average current density of 20 mA/cm^2 . Ni layer was initially electrodeposited on one side of the ribbon for 4 h and then on the other side for the same plating time. Thus laminated composite was produced in the form of a three-ply (Ni- $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ -Ni) laminated structure. Tensile test specimens were wire electrical discharge machined to have a gauge section of $10 \text{ mm} \times 3 \text{ mm}$. Tensile tests at room temperature were performed on an

Figure 5. The tensile fracture surface of the laminated composite containing $V_{\text{Ni}} = 0.77$ at different temperatures and with an initial strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$: (a) 450 °C, (b) 500 °C, and (c) higher magnification of Ni layers in (b).

Figure 6. Gas-pressure bulged domes of a $\text{Fe}_{78}\text{Si}_9\text{B}_{13}/\text{Ni}$ laminated composite.

Figure 7. The time-pressure curve.

Instron-CSS88000 machine with an initial strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$.

Tensile specimens were wire electrical discharge machined to have a gauge section of $10 \text{ mm} \times 3 \text{ mm}$. Tensile tests were performed using an Instron-CSS88000 machine in air atmosphere. They were carried out in the temperature range of 430–500 °C at different constant crosshead velocities. The tested specimens were heated at 30 °C/min from room temperature to the given temperature and held for 5 min for thermal equilibrium. Final elongation was measured in the gauge length in order to eliminate contribution from deformation in the specimen heads. For comparison, the tensile specimens of monolithic amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ ribbon and electrodeposited nano-Ni were also tested. The fracture morphology of the tested specimens was examined by scanning electron microscopy (SEM).

Bulging tests were carried out to evaluate the plastic forming properties of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}/\text{Ni}$ laminated composite. It was performed using the 600 kN universal testing machine, the bulging die is shown in Figure 1. The thickness of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}/\text{Ni}$ layered composite is 130 μm , the bulging temperature was set to be 450 °C, this is because the composite material has the best tensile properties at this temperature. The maximum bulging pressure is 4.0 MPa and holding time is 30 min. A good bulging part with RBH of 0.4 was obtained. The used forming medium was nitrogen gas.

3. Results and discussion

Figure 2 shows the micrographs of the $\text{Fe}_{78}\text{Si}_9\text{B}_{13}/\text{Ni}$ laminated composite. It is in the form of an $\text{Ni-Fe}_{78}\text{Si}_9\text{B}_{13}\text{-Ni}$ laminated structure. The thickness of each Ni layer is about 50 nm, and V_{Ni} in the laminated composite is 0.77. Figure 2a also

indicates the formation of a good bonding between amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ ribbon and electrodeposited Ni. In fact, the bend tests on the laminated composite show no evidence for delamination after about 180 °C bending. The Ni layers electrodeposited on continuous amorphous ribbon have a columnar grain structure in Figure 2b. The average size of Ni grains is about 50 nm.

Tensile specimens of the laminated composite containing $V_{\text{Ni}} = 0.77$ were tested in the temperature range of 430–500 °C and at an initial strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$. Figure 3 shows the specimens before and after the deformation compared to nano-Ni tested specimen. There is obvious uniform deformation in the gauge section without localized necking. In the selected temperature range, the largest elongation of 115.5% was obtained at 450 °C. It indicates good plasticity of $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ /Ni laminated composite. This elongation of the laminated composite is much larger than that of monolithic amorphous $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ ribbon (36.3%) and smaller than that of electrodeposited nano-Ni (276.5%) in monolithic form. The oxidation of the specimen surface becomes evident since the specimens were performed in air without gas protection.

Figure 4 shows true stress-strain curves under different temperatures at the strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$. Figure 5a shows the fracture surface of the gauge section deformed at 450 °C and an initial strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$. The vein-like structure morphology is characterized in $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ layer, which indicates $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ alloy keeps amorphous state. The vein pattern is quite different from that at room temperature and the ridges between voids are much higher. Most Ni grains maintain equiaxed shape and their average size is about 1.5 nm. The vein-like structure disappears in $\text{Fe}_{78}\text{Si}_9\text{B}_{13}$ layer when tensile temperature increases to 500 °C as shown in Figure 4. It reveals amorphous layer significantly crystallizes below Tx. The change of tensile behavior is clearly indicated by the fracture surface morphology. Figure 4c shows that the shape and average size of Ni grains tested at 500 °C is similar to that tested at 450 °C. This is probably ascribed that the increased internal energy at 500 °C is mostly absorbed by amorphous layer.

Figure 6 shows a bulged dome, under the conditions of 450 °C, 4.0 MPa and 30 min. The dome bulging height is 4.0 mm and the RBH 0.4. The bulging surface is smooth, no visible cracks found in macroscopic, indicating the layered composite material has a better ability to resist necking and plastic formability.

Figure 7 shows the time-pressure curve. At an initial stage of the bulging the pressure rapidly rises to 4.0 MPa. After reaching the limit value of the dwell pressure, the pressure decreases slowly and then finally, goes down to 2.8 MPa.

4. Conclusion

Monolithic nano-Ni layers exhibit tensile superplasticity and monolithic amorphous layer has low ductility. A maximum elongation of 115.5% was obtained at 450 °C and at an initial strain rate of $8.33 \times 10^{-4} \text{ s}^{-1}$ containing $V_{\text{Ni}} = 0.77$, which indicates good ductility of the laminated composite. The amorphous layer controls tensile behavior of the laminated composite and its flow-stress maintains a relatively high value during the whole tensile process, which indicates the amorphous layer does not fracture early. Under the conditions of 450 °C, 4.0 MPa and 30 min, a good bulging part with RBH of 0.4 has been obtained. It is suggested that the layered composite material has a better ability to resist necking and has good plastic formability.

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