

## Effects of Glass-Forming Metallic Film on the Fatigue Behavior of C-2000 Ni-Based Alloy

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### ABSTRACT

Glass-forming  $Zr_{47}Cu_{31}Al_{13}Ni_9$  (in atomic percent) films of various thicknesses were deposited on the C-2000 Ni-based alloy substrate by magnetron sputtering. Four-point-bending fatigue tests were conducted on the above system with the coated surface on the tensile side. It has been found that both fatigue life and fatigue-endurance limit can be considerably improved, while the degree of fatigue resistance enhancement depends on the maximum applied stress and the film thickness. Mechanisms of fatigue-resistance enhancements of the coated Ni-based alloy are discussed from the following aspects: reduction of surface roughness by the thin-film coating, good adhesion between thin film and substrate, development of residual compressive stress, and excellent ductility of glass-forming thin film (which would be otherwise brittle in bulk form). Of particular interest, we examine the interaction of substrate slip bands and the thin film ductile property, which would delay fatigue crack initiation process and thus extend the fatigue life.

### INTRODUCTION

Bulk metallic glass (BMG) materials show enhanced plasticity behavior when the propagation of shear bands is confined in order to prevent catastrophic failure, which thus motivates us to use a glass-forming metallic thin film as a coating material with anticipated high ductile properties. [1-6]. Thin films can be deposited on substrates as potential candidate materials for Micro-Electro-Mechanical Systems (MEMS) devices, owing to their excellent mechanical properties and high corrosion resistance [3]. The application of  $Zr_{75}Cu_{19}Al_6$  and  $Pd_{76}Cu_7Si_{17}$  glass-forming thin films as conical spring and micro actuator has been demonstrated [4-5]. By synthesizing amorphous layers on crystalline substrates, the surface performances could be improved due to high micro-hardness, low friction coefficient, and improved wear resistance [6]. Through thermal annealing of the as-sputtered  $Zr_{47}Cu_{31}Al_{13}Ni_9$  film in the supercooled liquid region ( $\Delta T$ ), the control of the nanocrystallization and amorphization could be realized [2]. Recently, people have discovered that the mechanical properties, especially the ductility of metallic glasses in the form of thin films, can be enhanced due to the geometry-confinement effects [7-10]. In view of their high strength, improved ductility, excellent wear and corrosion resistances, and good surface finishes, glass-forming films are anticipated to be ideal coating materials to improve the fatigue resistance behavior of structural materials.

### EXPERIMENTAL DETAILS

The HAYNES C-2000<sup>®</sup> Ni-based superalloy was used as the substrate material. The superalloy samples with geometry of  $3 \times 3 \times 25 \text{ mm}^3$  were ground with a 800-grit sand paper,

followed by electro-polishing to ensure a smooth surface. The bottom of the Ni-based alloy specimen was coated with the  $Zr_{47}Cu_{31}Al_{13}Ni_9$  (in atomic percent) glass-forming film by magnetron sputtering. Two sets of samples were prepared with film thicknesses of  $\sim 200$  nm and  $\sim 1$   $\mu\text{m}$ , respectively. Details of the film deposition are described elsewhere [2]. A computer-controlled Material Test System (MTS) servohydraulic testing machine was used for fatigue experiments. Samples were loaded at various stress ranges with an R ratio of 0.1 ( $R = \sigma_{\text{min.}} / \sigma_{\text{max.}}$ ; where  $\sigma_{\text{min.}}$  and  $\sigma_{\text{max.}}$  are the applied minimum and maximum stresses, respectively) under a load-control mode, using a sinusoidal waveform at a frequency of 10 Hz. In the four-point bending test, the distance between two supporting pins and that between two loading pins are 20mm and 10mm, respectively, and the maximum deflection of the specimen is about 2mm. The maximum strain is about 3%, and thus small deformation is assumed in our analysis. Upon failures, the samples were examined by SEM and AFM for fractography and surface-roughness investigations. TEM was used to characterize the film microstructure. The residual stress of the film was measured based on curvature change of the substrate before and after film deposition.

## DISCUSSION

Metallic-glass thin films deposited on the polycrystalline substrate are dense and uniform, as revealed in a typical TEM plan-view micrograph in Figure 1(a). The micrograph and a corresponding diffraction pattern indicate the structure as amorphous, with dispersed nanocrystalline phases in the size range of 10 - 30 nm. Figure 1(b) shows the SEM image of the as-sputtered thin film. From the smooth and featherless contrast, it can be realized that the film deposition can improve the surface finish of the substrate. Figure 2 shows the AFM images of the uncoated and coated materials. Apparently, the rms surface roughness decreases from 20.62 nm to 15.53 nm after the application of the 200 nm-thick coating, and further down to 12.17 nm with the 1  $\mu\text{m}$ -thick coating. Therefore, the surface roughness of the coated Ni-based alloy is reduced, which may decrease the stress concentration sites and increase the fatigue resistance of the coated sample. Detailed discussion of fatigue crack initiation will be presented shortly.

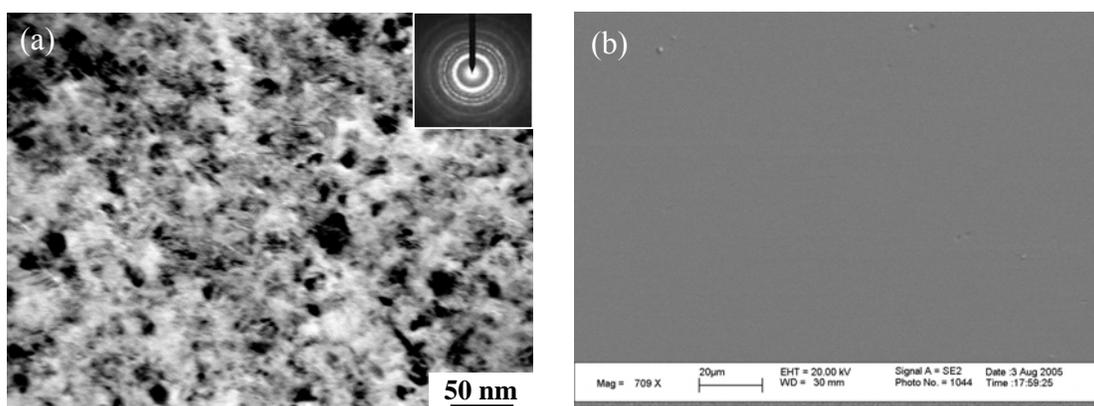


Figure 1. (a) Plan-view TEM micrograph and diffraction pattern (inset) of the glass-forming  $Zr_{47}Al_{31}Cu_{13}Ni_9$  film, and (b) SEM image of the as-sputtered thin film.

Figure 3 shows the fatigue experimental results of the coated samples along with those of the

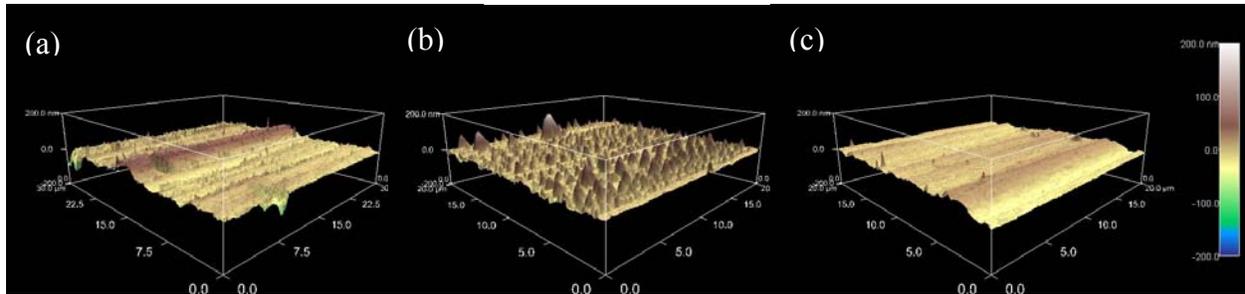


Figure 2. AFM topography images of (a) Ni-based alloy with rms roughness of 20.62 nm, (b) Ni-based alloy coated with 200nm-thick film and rms roughness of 15.53 nm, and (c) Ni-based alloy coated with 1 $\mu$ m-thick film and rms roughness of 12.17 nm.

uncoated samples. At high stress levels, almost no beneficial effect of the coating on the fatigue life can be seen. Improvement of the fatigue lives becomes more pronounced, as the applied stress decreases. The fatigue life can be increased by 474% for the 200 nm-thick film, and 108% for the 1  $\mu$ m-thick film, when tested under maximum stress of 600 MPa. The fatigue life at the same stress level and the fatigue-endurance limit of the alloy coated with 200 nm-thick film are slightly higher than those of the alloy coated with 1  $\mu$ m-thick film.

Figure 4 (a) shows the SEM fractograph of the 200 nm-thick coated sample tested at  $\sigma_{\max.} = 725$  MPa. The whole fracture surface consists of three regions: the fatigue-crack-initiation, crack-propagation, and final-fast-fracture areas. The fatigue crack seems to initiate from the corner of the coated side and to propagate inward. Figure 4(b) presents a SEM micrograph revealing the interface of the film and the substrate. It is clear that the film remained relatively well adhered with the substrate after the final fatigue fracture, without observable cracks on the film. The good film adhesion with the substrate suggests that the enhanced ductility of the thin film can accommodate the deformation during the fatigue test under high stress levels.

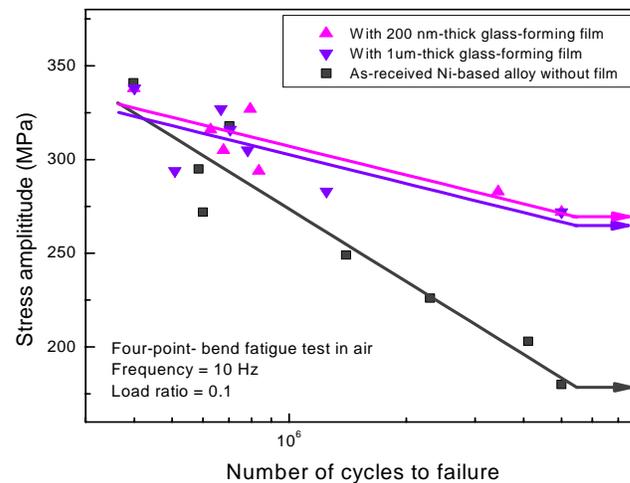


Figure 3. Stress vs number of cycles to failure (S-N) curve of the Ni-based alloy with and without Zr-based glass-forming films.

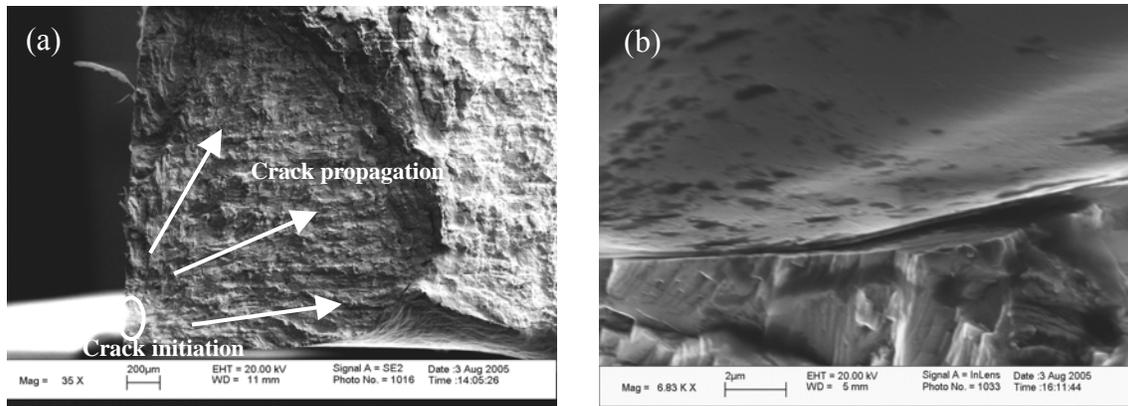


Figure 4. Fractography of the fatigued specimen with a 200 nm-thick film tested at  $\sigma_{\max.} = 725$  MPa, (a) the whole fracture surface, and (b) the fracture surface near the film.

Residual stresses in the surface film can readily be developed during the film deposition process. The residual stress of the 200 nm-thick film was measured to be compressive, around 80 MPa. Compressive residual stresses are beneficial to increase the fatigue resistance of the coating, because it prevents crack nucleation from the surface [11]. However, the compressive residual stress in the current study is much lower than that in some hard coatings as TiN or TiC, indicating that the residual stress of the present study may not play a major role in affecting the fatigue behavior.

Figure 5(a) shows the inclined side of the four-point-bending sample with the amorphous thin film. Typically, a large number of slip bands will propagate to the surface and form extrusions/intrusions, which may serve as the crack-initiation sites. However, amorphous thin films bonded to the tensile side will block the dislocation pileup and the slip deformation, and therefore the fatigue crack initiation is retarded. The reason for the coating to accommodate substrate slips and to reveal enhanced ductility will be addressed in Fig. 6 shortly. Figure 5(b) shows the slip bands traveling to the compression side. It is obvious that without the shielding effect from the thin film, the slip deformation is easy to propagate to the surface and cause surface offsets.

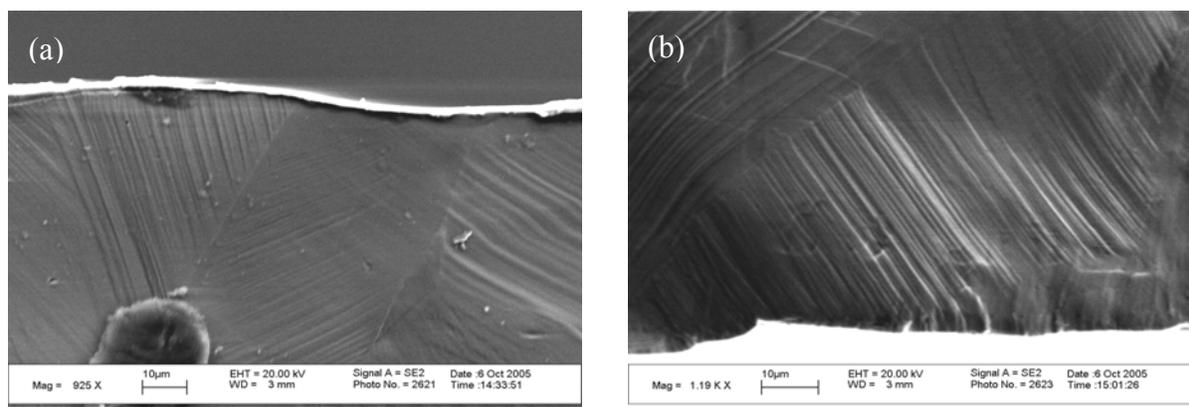


Figure 5. The slip traces appeared in the inclined side without the amorphous thin film, (a) near the tension side, and (b) near the compression side.

In comparison to the amorphous thin ribbons or films, the fatigue lives of the amorphous

thin film in the present study are much longer [12]. Compared with the fatigue-resistance improvement with other coatings, the enhancement of the fatigue lives in this research is very significant [13-14]. Then, what is the reason for the remarkable fatigue-resistance improvement with such a thin film? In the fatigue tests of amorphous thin ribbons or sheets, an incubation period elapsed before a fatigue crack started [13]. Fatigue results of the amorphous Ni-P thin films show that the fatigue life is dominated by crack initiation. Once a fatigue crack starts to grow, the fatigue fracture occurs only after several thousands of cycles [3]. For the present study, the fatigue-resistance enhancement is mainly due to the lifetime extension that occurs during the fatigue crack-initiation stage. Once the fatigue crack is formed, it will propagate through the thin film to the substrate or the interface between the film and substrate. Thus, from the observations in Figure 5, a hypothetical fatigue mechanism of the fatigue-crack initiation and propagation behavior in the amorphous thin film is proposed, as shown in Figure 6. Dislocation pileups or persistent slip bands, when arriving at the surface, can create the surface offset, which serves as the potential fatigue-crack-initiation site. The amorphous thin film can prevent the surface offset because of its high ductility and strength, yielding an improved fatigue property. The enhanced ductility of the glass-forming metallic thin film is due to the localized slip and the localization of shear bands in the thin film, thus preventing catastrophic failure by single-shear-band propagation and promoting thin film ductility.

The beneficial properties of the coating, such as the higher mechanical strength and the existence of a residual compressive stress within the coating, can only be transmitted to the substrate through good adhesion of the film to the latter. When the film is under a state of high compressive stress, a reduction in the adhesion of the film to the substrate may occur [15], which may explain why the surface roughness of the 1  $\mu\text{m}$ -thick film is low, yet its fatigue life under the same stress level and the fatigue endurance limit is slightly lower than the 200 nm-thick film specimen. As shown in Fig. 6, the slip band tends to create surface offset and a potential delamination site. Whether a thick or thin film can prevent decohesion also depends on the thin film ductility. So far, we have not measured the work of adhesion of the interface, although no evidence of delamination in Fig. 4 suggests good adhesion. The interaction of slip bands, BMG thin film ductility and the interface adhesion will be amenable to experimental study.

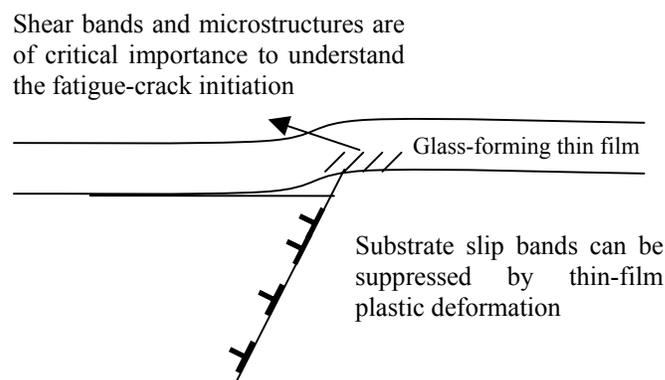


Figure 6. A hypothetical mechanism showing fatigue-crack initiation (typically due to dislocation pileups and surface offsets) suppressed by the unique inelastic deformation of glass-forming films.

## CONCLUSION

With the deposition of the  $Zr_{47}Cu_{31}Al_{13}Ni_9$  glass-forming film, the fatigue life and fatigue-endurance limit of the C-2000 Ni-based superalloy could be remarkably improved. The fatigue-resistance enhancement is mainly due to the suppression of the fatigue crack-initiation process. The good adherence of the film to the substrate, and the improved surface finishes of the film have noticeable influence on the fatigue life. The high strength and the good ductility might be other important factors for the improvement, while the small compressive residual stress plays a minor role for the enhancement. Therefore, the present research can provide an effective means to improve the fatigue resistance of materials and components using the glass-forming films.

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