# Interfacial microstructure and fracture behaviour of Fe/Ni interface by solid-state compressive bonding

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Interface is pivotal in property improvement in layered structures, whereas its multiple factors at various length scales restricted further investigation. In this study, we focused on the effects of interfacial microstructure on interfacial strength in Fe/Ni interface. It was found that the bicrystal interfaces without pre-exist grain boundaries (GBs) exhibit relatively higher interfacial crack initiation strength. In-situ observation revealed that the GB-interface junctions have relatively lower intrinsic bonding strength and behaved as weak spots in tensile test.

Keywords: interface, compressive bonding, fracture behaviour, interfacial property, multi-scale study

## 1. Introduction

Multilayered Steels (MLSs) have attracted considerable attention owing to their unique advantage of combining impressive strength and ductility <sup>1</sup>). Many studies reported that interfaces between layered components are pivotal for overall property improvement <sup>2, 3</sup>). In specific, strong interfaces improve the strength-ductility combination, whereas relatively weak interfaces are preferred to enhance the overall toughness <sup>3, 4</sup>). Therefore, it is necessary to clarify the role of the interface in layered structure for further property improvement. However, it has been indicated that interfacial fracture is a complicated deformation process simultaneously at various length scales, and their concurrent effect restricts further investigation of each factor individually.

At macro scale, interfacial configurations such as voids and roughness variation significantly influence interfacial adhesion. Moreover, strength evolution of the bonded part has been shown with the primary responsibility for strength improvement <sup>5)</sup>. At micro scale, interfacial microstructure plays a vital role in interfacial adhesion. In specific, deformation-induced Dynamic Recrystallization (DRX) at interface and Grain Boundaries (GBs) in the vicinity are expected as potential factors. However, their mechanisms have yet to be clarified. At atomic scale, interfacial fracture is intrinsically the debonding of the interfacial atoms. Work of adhesion quantifies the energy required to break the adhesion, where interfacial energy is widely accepted as the most important variable of it.

So far, many studies have focused on interfacial property improvement, whereas the pre-exist GB effects on interfacial adhesion have not been clarified. It is necessary conduct a multi-scale study on GB-interface interaction and hence, to clarify its contribution to overall interfacial properties. In this study, solid-state compressive bonding method is applied to precisely control the interfacial microstructure. The main objective is to investigate the effect of pre-exist GBs on interfacial properties and fracture behaviour in Fe/Ni interfaces.

#### 2. Experiments

IF steel (0.006C-0.2Mn-0.038Ti-Fe) and pure Ni (99.5 wt.%) were used as the component layers in this study.

Cylindrical samples were heat treated at  $1250^{\circ}$ C for 24 hours, followed by a cooling rate of  $1^{\circ}$ C/min. This is to enlarge the average grain size. The bonded surfaces were mechanically polished to eliminate the surface roughness effect on the interface. Subsequently, pairs of IF steel and Ni samples were joined by the solid-state compressive bonding method with a bonding pressure of 50 MPa and temperature of 500°C. Compressive time was controlled from 5 s to 100 s to fabricated specimens with weak and strong interfacial adhesion. Then the as-compressed samples were cooled to room temperature with a cooling rate of  $3^{\circ}$ C/s.

After the bonding process, the cylindrical specimens were cut precisely into either bicrystal or polycrystal interface specimens. The bonded cylindrical specimens were first cut into thin sheets. To confirm the GB distribution near the interfaces, OM observation and EBSD analysis were conducted on both sides of the surfaces. Then the sheets were further cut into bar specimens by precisely selecting the cutting positions. The interfacial size of all cut specimens was about 0.8 mm  $\times$  0.6 mm, and a notch was introduced to each interface to ensure the fracture initiates from the interface.

Uniaxial tensile tests were conducted at room temperature to evaluate the interfacial property evolution with a constant strain rate of  $1.5 \times 10^{-4}$  s<sup>-1</sup>. More than three measurements were performed for each condition to ensure the reliability of the experimental results. In-situ observation was conducted by confocal laser microscopy (1LM15, LASERTEC) to investigate the interfacial fracture behaviours.







Before Tensile Test

After Crack Initiation

Fig. 2. EBSD and SEM observation on the 40 s bicrystal interface (a) before tensile test and (b, c) just after crack initiation.

### 3. Results

# **3.1 Interfacial property evolution**

Figure 1 shows interfacial crack initiation strength evolution per contact area with compressive time. In general, interfacial crack initiation strength exhibited a two-stage growth pattern with increasing compressive time. Moreover, the bicrystal interfaces exhibited slightly higher interfacial crack initiation strengths compared to the polycrystal interfaces. This indicates that the bicrystal interfaces showed higher interfacial fracture resistance. It is noteworthy that the only difference between the bicrystal and polycrystal specimens was whether the pre-exist GBs were inclusively cut or not. It is inferred that the pre-exist GBs negatively impact the interfacial adhesion.

## **3.2 Fracture behaviour**

In-situ observation indicated distinct interfacial fracture behaviours in this study. In bicrystal interfaces, crack initiated from the notch-interface spot owing to the local stress concentration. Subsequently, crack propagation from the notch-interface spot along the interface was developed, forming a complete fracture. Additionally, slip bands with shallow patterns were observed directly from the deboned interface. The uniformly distributed patterns indicated that there is no unique local behaviour at the bicrystal interface.

Compared to the bicrystal interfaces, the polycrystal interfaces exhibited a distinct crack initiation behaviour. Figure 2 indicates the relationship between interfacial microstructure and crack initiation behaviour at the 40 s polycrystal interface. SEM observation on the interface just after crack initiation is shown in Fig. 2(b), where the interfacial crack initiated from the right pre-exist GB-interface junction, the closest junction to the notch. This implies that both the notch-induced stress distribution and pre-exist GB effects influenced the crack initiation behaviour in polycrystal interfaces. Additionally, the slip bands from the pre-exist GB demonstrated that the interfacial crack introduced higher strains to the adjacent grains. Instead of generating directly from bicrystal interfaces, the polycrystal slip bands grew from the crack-connected GB, propagated to inner parts, and then impeded by other GBs. This implies that the crack-induced torque was dissipated by intragranular slip. Furthermore, the various slipping systems in adjacent grains promoted slip deformations along different directions. As a result, the mobility difference between the two adjacent grains led to local deformation differences at the GB-interface junction.

#### 4. Discussions

Overall, the fracture behaviour in polycrystal interfaces indicated that the pre-exist GB-interface junctions behaved as weak spots in tensile fracture by providing potential crack initiation sites. This demonstrates why the bicrystal interfaces generally exhibited relatively higher interfacial crack initiation strength than the polycrystal interfaces. To explain the polycrystal crack initiation phenomenon, there are two possible influencing mechanisms: local stress concentration at the GB-interface junctions and relatively weak intrinsic bonding strength at the junctions.

At atomic scale, interfacial dislocations locate all along the bonded interface and contribute to the local stress redistribution <sup>6)</sup>. The intragranular parts show much higher mobility, whereas GBs hinder dislocations and form pile-ups. This resulted in decreased local bonding strength. At micro scale, the intragranular slips were impeded by the GBs, which can be evidenced by the slip bands. The pre-exist GBs conjugated distinct slipping behaviours in adjacent grains, leading to local stress concentrations.

# 5. Conclusion

A multi-scale study was conducted on the effect of interfacial microstructure on strength evolution in Fe/Ni interfaces. The pre-exist GBs were indicated with negative impact on the interfacial strength. This related to the unique crack initiation behaviour at the GB-interface junctions. Relatively lower bonding strength and stress concentration take the main responsibility.

## References

- T. Koseki, J. Inoue and S. Nambu: Mater. Trans. 55, 2 (2014) 227-237.
- J. Park, C. Jo, T. Song, H. Kim, S. Sohn and S. Lee: Mater. Sci. Eng. A 727 (2018) 320-328.
- S. Nambu, M. Michiuchi, J. Inoue and K. Koseki: Compos. Sci. Technol. 69, 11 (2009) 1936-1941.
- M. Pozuelo, F. Carreno and O. Ruano: Compos. Sci. Technol. 66, 15 (2006) 2671-2676.
- 5) K. Pongmorakot, S. Nambu, Y. Shibuta and T. Koseki: Sci. Technol. Weld. Join. 22, 3 (2017) 257-263.
- L. Zhang, Y. Shibuta, C. Lu and X. Huang: Acta Mater. 173 (2019) 206-224.