



Evaluation method for the interfacial strength of thermally grown Al_2O_3 scale and Ni-Al alloy

Chihiro Tabata^{a,c,*}, Taiyo Maeda^b, Thomas Hoefler^a, Shingo Ozaki^b,
Kyoko Kawagishi^{a,c}, Shinsuke Suzuki^c, Takahito Ohmura^a, Toshio Osada^{a,b}

^a National Institute for Materials Science, Ibaraki, Japan

^b Yokohama National University, Kanagawa, Japan

^c Waseda University, Tokyo, Japan

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ABSTRACT

Nanoindentation techniques are useful for evaluating the critical load for crack initiation on interface between Ni-Al alloy and thin surface Al_2O_3 scale formed at high temperature, since interfacial cracking can be clearly detected as a pop-in event. However, actual interfacial stress applied during loading and when cracked is unclear, due to the multiaxial stress field. Here we analyzed interfacial stress distribution during the nanoindentation using finite element analysis. Elastoplastic simulations of nanoindentation were performed with local stress-strain curves for oxide scale and the Al depletion zone. Local stress-strain curves were inversely analyzed from experimental load-penetration depth curves, obtained using separate nanoindentation tests. Interfacial stress was obtained as a function of indentation load, clarifying that shear stress acting on interface contributed significantly to crack initiation. The two-parameter Weibull plots of interfacial strength $\sigma_{\text{Int,max}}^c$ were converted from pop-in load P_c obtained in experimental nanoindentation tests for samples with different amounts of impurity S segregation. Largest differences appear where fracture probability F is low; for $F = 4\%$, difference of approximately 780 MPa between the sample with higher S segregation level (high $S_{\text{interface}}$ alloy, $\sigma_{\text{Int,max}}^c = 1.73$ GPa) and lower S segregation level (low $S_{\text{interface}}$ alloy, $\sigma_{\text{Int,max}}^c = 2.51$ GPa) were obtained.

1. Introduction

Ni-base superalloys have excellent oxidation resistance; however, ppm level impurity S is known to drastically decrease the adhesion of the protective Al_2O_3 scale [1–6], resulting in degradation of cyclic oxidation performance of single crystal turbine blades. This is due to the segregation of S impurities at the interface of the thermally grown Al_2O_3 scale and the Ni-base alloy [7–13], where amorphous-like intermediate layers with high level of S exist [14]. This is said to lower the adhesion between the Al_2O_3 scale and the alloy, since according to several first-principal density functional theory calculations, the existence of S prevents the Al and O atoms from forming strong bonds [9,10,14]. Macroscopic experimental methods such as tensile pull tests [4,11] and scratch tests [15,16] have been used to assess the effects of S segregation on the adhesion of the oxide scales; however, large deviations were observed especially for thin and multi-layered oxide scales. Therefore, methods to quantitatively and directly assess the bond strength at the interface, in

relation to the S content and other elements that may affect the adhesion or failure strength, are needed for understanding and simulating the cyclic oxidation performance of superalloys in the actual environment.

Cross-sectional nanoindentation methods have been proven useful to assess interfacial adhesion and delamination for different kinds of materials [17–22]. In our previous study, we measured the thin Al_2O_3 surface scale/Ni-Al alloy critical load for crack initiation at interface using cross-sectional nanoindentation [23]. This confirmed that the initial pop-in load related to interfacial cracking for Ni-Al alloy decreases with increasing S segregation level. However, nanoindentation only provides information about the load, and cannot evaluate the actual stress acting at the interface. In addition, the hardness of alloy is generally lower than that of an oxide scale, so the indenter pressed into the scale tends to move toward the softer alloy. These made it difficult to understand how the cracks had initiated, since in-situ observation of the nanoindentation tests cannot be conducted due to the setup of the machine. Further, actual interfacial stress applied during loading and cracking is not

* Corresponding author.

E-mail address: tabata.chihiro@nims.go.jp (C. Tabata).

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clearly understood, because of the multiaxial stress field on the sample.

To directly evaluate the interfacial stress and strength, an approach using numerical simulation could be useful. Previous research commonly used finite elemental analysis (FEA) to assess the nanoindentation test results [24–28]. Inverse estimation techniques are proposed by several research groups, which incorporate the load-penetration depth curve obtained from the nanoindentation testing and analyzed 3D nanoindentation marks, to obtain the mechanical properties of the materials [29–31]. The combination of these inverse estimation techniques and FEA are often conducted to determine the mechanical properties of materials and coatings [32,33]. For example, Reuther et al. used reverse FEA approach based on nanoindentation, to reproduce their experimental findings and obtain adhesion model parameters, such as critical adhesion energy [34]. Yang et al. also used cohesive constitutive model to reproduce the SiC/SiO₂ interfacial delamination [35]. Therefore, FEA is effective for understanding the crack initiation process at the interface during the nanoindentation test and obtaining the interfacial strength. However, this method has not been implemented for analyzing the interfacial strength of the thermally grown oxide layer and Ni-base alloys. Additionally, detailed analyses of oxidized Ni-base alloys and the mechanical properties of the Al depletion zone that forms after oxidation have not been obtained before. Hence, this work is essential for becoming one step closer to the understanding and quantification of interfacial strength of thermally grown oxide layers.

The purpose of this study is to propose a method which helps clarify the stress distribution during nanoindentation using FEA and assess the interfacial strength when cracked. The parameters of elastoplastic properties for the alloy and oxide scale were determined through previously proposed inverse analyses of nanoindentation. Using the model, comparison with the nanoindentation test results from the experiment [23] and analysis on where the interfacial crack propagation had initiated were conducted. While comparing with previous studies that assessed the interfacial strength using failure stress [4,11], the values of normal stress and shear stress at the interface of the Al₂O₃ scale and Ni-Al alloy were obtained.

2. Experimental and evaluation methods

2.1. Procedures for obtaining elastoplastic properties using nanoindentation

The Ni-9.8wt.%Al sample with lower amounts of interfacial S segregation (low S_{interface} alloy, prepared using CaO crucible) oxidized at 1100 °C for 1 h, used in our previous study [23], and a high-purity (99.99 %) α-Al₂O₃ powder (AKP-50, Sumitomo Chemical Co., Ltd) sintered at 1600 °C [36], were used to obtain the stress-strain curves incorporated in the FEA. The cross-section of the low S_{interface} alloy was observed using field emission scanning electron microscope (FE-SEM, GeminiSEM 300, ZEISS) at an acceleration voltage of 10 kV. Both secondary electron images and back-scattered images were taken by 1024 × 768 pixels for most of the images, while the images covering the large areas were taken by increasing the resolution to 12288 × 9216 pixels. The morphologies of the oxide layers for the low S_{interface} alloy, as well as the Ni-9.8wt.%Al sample with higher amounts of interfacial S segregation and oxidized at 1100 °C for 1 h (high S_{interface} alloy, prepared using Al₂O₃ crucible), also used in our previous study, were observed using scanning transmission electron microscopy with energy dispersive x-ray spectroscopy (STEM-EDS, FEI Titan G2 80–200) at an acceleration voltage of 200 kV. The samples were prepared using focused ion beam (FIB, FEI Helios NanoLab 650 and G4UX). Detailed explanation of the morphology of the oxide layers and alloy will be explained in section 3.

First, nanoindentation tests (TI 950 TriboIndenter, HYSITRON) were conducted using a diamond Berkovich indenter with an apex angle of 115 degrees for both samples. Here, the indentation was conducted using a separate pure Al₂O₃ sample, rather than the actual oxide scale,

due to the limited thickness of the scale and the difficulty of observing the pile-up within the oxide scale during the inverse estimation. Also, the apex angle of the indenter used for obtaining the elastoplastic properties are significantly larger compared to the indenters used in the cross-sectional nanoindentation in our previous study [23]. Nanoindentation tests were conducted at the load of 10, 20, and 25 mN for the Al₂O₃ sample, and the results where the indentation marks were located within the grains and showed no signs of cracking were selected. Nanoindentation tests for the Ni-Al sample were conducted starting at the Al depletion zone to the γ/γ′ two-phased region at a constant load of 5 mN. 100 nanoindentation tests (5 columns × 20 points: each column 60 μm apart, each indentation 2 μm (x-axis) and 15 μm (y-axis) apart) were conducted diagonally in these regions. This was conducted to calculate the Young's modulus and hardness, using the techniques proposed by Oliver and Pharr [37,38]. Results with the clear load-penetration depth curves and indentation marks were used for the inverse analysis, explained in the next section.

Secondary electron and back-scattered images of the Ni-Al sample were taken at the regions where the nanoindentation tests were conducted, to obtain the γ′ area fraction. The microstructures were obtained and analyzed automatically using FE-SEM, over an area of (340 × 42 μm²), avoiding the nanoindentation marks as much as possible. The γ′ area fraction was obtained using an automated evaluation program, the same method proposed by Hoefler et al. [39]. Point analyses using the electron probe micro analyzer (EPMA, SHIMADZU EPMA 1610) at a beam size of 3 μm at 15 kV, 20 nA were conducted in these areas as well. 100 points (5 points per row × 20 rows), starting from the Al depletion zone to the γ/γ′ two-phased region were taken in between the columns of the nanoindentation tests. This was conducted for all four areas in between the five columns of nanoindentation tests, to analyze the changes in the Al content, in relation to its elastoplastic properties.

2.2. Inverse analysis method for stress-strain curve

For both materials, the reduced Young's modulus E_r and the nanoindentation hardness H were calculated from the load-penetration depth curve obtained from the experimental nanoindentation tests using the Oliver-Pharr method [37,38]. Here, the loading and deformation behavior satisfies the Kick's law [40–43], $P = Ch^2$, where P is the applied load, C is the curvature of load-displacement curve during loading, and h is the penetration depth of the indenter. The hardness H of the material is determined from the experiment by the equation, $H = \frac{P}{A_c}$, where A_c is the contact area at $h = h_c$, which is the contact depth of the indenter. Typically for the Berkovich indenter, this is approximately $A_c = 24.5h_c^2$ [44,45]. The E_r was calculated from the load-penetration depth curves by the equation, $E_r = \frac{S\sqrt{\pi}}{2\sqrt{A_c}}$, where S is the contact stiffness.

The inverse analyses of the nanoindentation marks to obtain the elastoplastic properties were conducted, using the same method presented by Hoefler et al. [39]. First, the topographic information of the indentation mark was obtained through automated analysis using FE-SEM. The depth of the indentation mark and the height of the pile-up were measured by taking four angular selective backscattered electron (AsB) images of the indentation, one for each channel. The normalized pile-up height H_p was calculated using the equation below:

$$H_p = \frac{\sum_{k=1}^3 h_{p(k)}}{3 h_{\max}} \quad (1)$$

where $h_{\max}$ is the maximum indent depth and $h_{p(k)}$ is the maximum pile-up height obtained from the three lines k drawn from the indent corners through the indent center. The Young's modulus E of the sample was calculated using the relation $\frac{1}{E_r} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu^2}{E}$, where the E_r was obtained for each nanoindentation test. Here, the Poisson ratios for the Ni-Al alloy and the Al₂O₃ sample were $\nu = 0.31$ and $\nu = 0.24$ [46,47], respectively, while the Young's modulus and Poisson's ratio for the indenter were E_i

= 1140 GPa and $\nu_i = 0.07$, respectively. Assuming the proportionality of H to the representative stress σ_r ,

$$\sigma_r = \frac{H}{k_e} = A + B\varepsilon_r \quad (2)$$

where A is the intercept representative strain ε_r of around 8 %, and B is the work hardening rate. Assuming linear strain hardening, the yield stress can be expressed using the equation below:

$$\sigma_y = \frac{A}{\left(1 - \frac{B}{E}\right)} \quad (3)$$

In addition, the stress–strain curves can be calculated from the nanoindentation results by the following equation:

$$H_p = k_{p1} \exp\left(-\frac{k_{p2}\sigma_y + k_{p3}B}{E}\right) \quad (4)$$

The parameters used in eq.2 and 4 are shown in Table 1. Using eq.1 to 4, the work hardening rate B and yield stress σ_y were obtained.

After obtaining these parameters, the respective equations for the elastic and plastic regions were used to create stress–strain curves:

$$|\sigma| = E \times |\varepsilon| (\varepsilon_p = 0) \quad (5)$$

$$|\sigma| = \sigma_y + B|\varepsilon_p| (\varepsilon_p \geq 0) \quad (6)$$

Here, σ represents stress, E is the Young's modulus, ε is strain, and ε_p is plastic strain. Note that the actual sample used in the previous experiment was about $4 \times 10 \times 5 \text{ mm}^3$ in size, with oxide scales of about 3 μm in width. Although the actual sample has several different oxide scales, the simulation uses only the values of the Al_2O_3 scale. This was (i) to simplify the model and (ii) due to the lack of data and difficulty obtaining the parameters experimentally for the complex oxide scales that form on top of the Al_2O_3 scale.

2.3. FE model for Al_2O_3 scale and Ni-Al alloy

The elastoplastic deformation during nanoindentation was simulated with the commercial FEA software package, LS-DYNA R11.0.0 [48], using the dynamic explicit method. The results in this paper only simulate up to the pop-in event observed in the load–penetration curves, because we focused on the state at the instant of crack initiation. Fig. 1 shows (a-1) top and (a-2) front view of the FE model of the sample, and (a-3) the indenter used for this research. The model consists of an Al_2O_3 surface oxide scale and a Ni-Al alloy, reproducing the experiment shown in Fig. 1(b) [23]. The analysis targets the region near the indenter tip, measuring $7.17 \times 10.24 \times 4.16 \text{ }\mu\text{m}^3$. To reduce computational cost, a y-symmetric half-model was employed. The sample consists of hexahedron elements, with the smallest mesh size being 8.9 nm in length, located around the indenter contact region. This mesh size used in this study is comparable to, or smaller than those used in previous studies that performed mesh convergence to ensure the accuracy of the simulations [49–54]. A bilinear isotropic hardening model was adopted for both the Al_2O_3 scale and the Ni-Al alloy constituting the sample. Nodes on the Al_2O_3 scale/Ni-Al alloy interface were linked via tied connection, enabling direct acquisition of stresses acting on the interface. Hexahedron solid elements are used for the mesh under the indenter. Although a formal mesh sensitivity or convergence study was not conducted, the selected mesh sizes under the indenter were sufficiently small to prevent

Table 1
Parameters used in the inverse analysis.

ε_r	k_e	k_{p1}	k_{p2}	k_{p3}
0.08	7.23	1	0	229.1

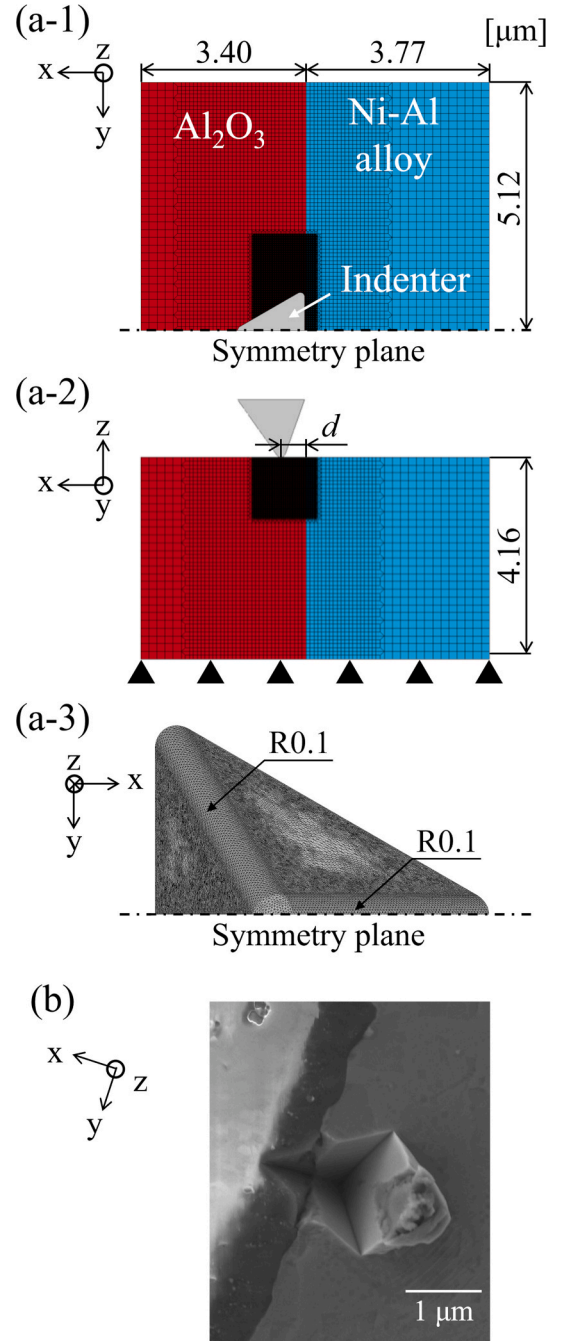


Fig. 1. The (a-1) top view and (a-2) front view of the FEA model of the sample and (a-3) indenter. (b) SEI of the nanoindentation mark at the interface of the Al_2O_3 scale and Ni-Al alloy from the cross-sectional nanoindentation test conducted in previous research [23]. The indenter was free in the x-direction, and the bottom surface of the sample was fixed in the z-direction displacement.

penetration of the indenter into the elements.

The 60-degree indenter consists of tetrahedron elements, and the tip of the indenter has a fillet radius of 0.1 μm . The indenter was constructed as a rigid body, with the assumption that its deformation is negligible. The indenter was placed in four locations with different initial distance d from the tip of the indenter to the interface: $d = 0.10, 0.35, 0.50, 0.65 \text{ }\mu\text{m}$. Although the cross-sectional nanoindentation tests were conducted at $d = 0.50 \text{ }\mu\text{m}$ [23], due to the encoder resolution of the nanoindentation system being 100 nm and the micro-stepping resolution being 50 nm [55], various distances were simulated to incorporate the errors. At the initial state, there is a slight distance between the sample

and the indenter.

The experiments were conducted under load control [23], but the analyses were performed under displacement control for computational stability. It is important to emphasize that this study only simulated up to the pop-in event. Up to this point, no cracks have initiated, and only elastoplastic deformation occurs. Therefore, the use of cohesive element model is not required. The indenter was free in the x-direction and pressed into the z-direction at a constant rate to a penetration depth of 470 nm. The bottom surface of the sample was fixed in the z-direction displacement. The friction coefficient between the sample and the indenter was set to 0.03, and the penalty method was used to treat the contact conditions. Since the present analysis is quasi-static, mass scaling was employed to reduce computational time. It was confirmed that the ratio of kinetic energy to internal strain energy in the FE model remained sufficiently small. This simulation was conducted under the assumption that the pop-in event represents the crack initiation at the interface, and that the crack formation at the interface will occur before fracture within Al_2O_3 scale, which has been assessed in our previous study [23]. Although the thermal residual stress has been neglected in this simulation, which is for future work, several factors were taken into consideration in this work. For example, the cross-sectional nanoindentation tests were conducted so that the indenter is parallel to the interface, to avoid the effects of the interfacial roughness.

3. Results

3.1. Stress-strain curves for each phase

First, the elastoplastic properties of the Ni-Al alloy were obtained. Fig. 2 shows the (a-1) secondary electron image and (a-2) backscattered image of part of the Ni-Al sample where the nanoindentation tests were conducted, and the EPMA analysis and the γ' area fraction were obtained. Although the SEI seems as though there are several grains, the crystal orientation of the sample is mostly (100). The red squares in Fig. 2(a-2) show where the AsB images of (b-1) Al_2O_3 scale and nanoindentation marks at the (b-2) Al depletion zone and (b-3) γ/γ' two-phased region were taken. The dark rectangular phase is the γ' , while

the bright channel is the γ phase. The amount of γ' phase is clearly lower in the Al depletion zone, compared to the γ/γ' two-phased region. This is due to the lack of Al in this region, caused by the oxidation and formation of the Al_2O_3 scale.

The changes in the γ' area fraction (blue) and Al concentration (black) in relation to the distance from the boundary of the Al depletion zone and γ/γ' two-phased region are shown in Fig. 3(a). Here, the error bars represent the standard deviation of the γ' area fraction and Al concentration, respectively. The x-axis represents the distance from the boundary of the Al depletion zone and γ/γ' two-phased region, seen in Fig. 2(a-1, a-2). For the γ' area fraction, this was determined by obtaining the distance from the middle of each secondary electron image (SEI, $7 \times 10 \mu\text{m}$) to the boundary and taking the average for each row. As for the Al concentration, because these point analyses were set up to align with the nanoindentation marks, the average distance from the boundary for the five nanoindentation marks in the same row were used. Clear changes in the γ' area fraction can be seen in both the SEI and the measured results, due to the formation of the Al depletion zone through oxidation. This can be confirmed by the clear decrease in the Al content in the same areas where the γ' area fraction was low. Fig. 3 also shows the changes in the (b) Young's modulus E and (c) hardness H obtained from the Ni-Al sample, in relation to the distance from the boundary between the Al depletion zone and γ/γ' two-phased region. The white plots represent the results for the nanoindentation tests where the indentation marks were observed in the Al depletion zone, seen in Fig. 2(a-2). The colored plots represent the results obtained from the γ/γ' two-phased region. The dotted lines are the average values for each parameter obtained at the Al depletion zone, while the dashed lines are of the γ/γ' two-phased region. The highlighted areas show the location of the undulating interface, in relation to the boundary. The dashed lines are the average distance of the location of interface from the boundary, while the dotted lines show the minimum and maximum distances. Here, the values of E were relatively similar between the Al depletion zone and the γ/γ' two-phased region. The values of E obtained through this method were similar compared to previous research that modeled this parameter through computational approach [56]. These values were also compared with calculations obtained by JMatPro (v16, Ni-

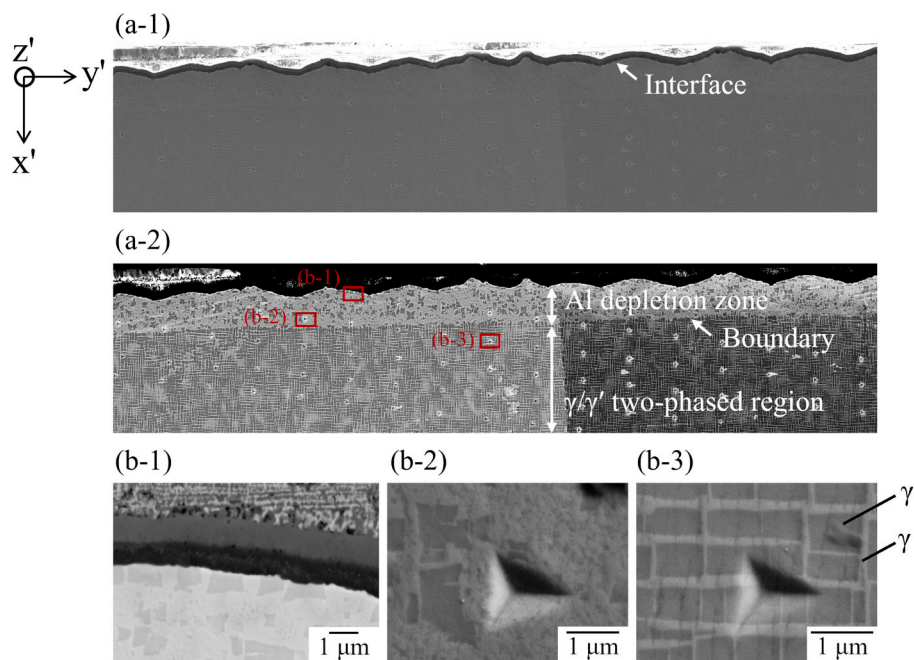


Fig. 2. Sample observation after nanoindentation tests. (a-1) SE and (a-2) AsB images of the Ni-Al alloy used for the nanoindentation tests. The locations indicated by the red squares are where the AsB images of the (b-1) Al_2O_3 scale and nanoindentation marks at the (b-2) Al depletion zone and (b-3) γ/γ' two-phased region were taken. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

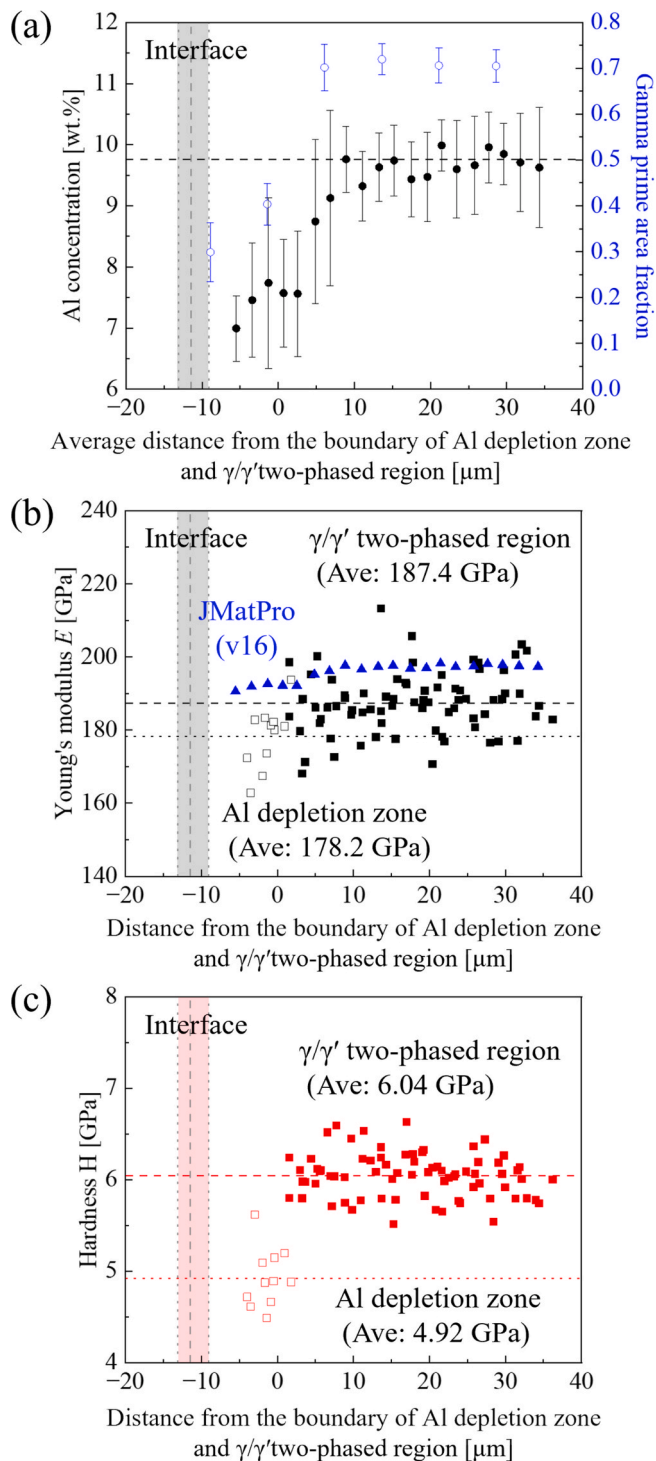


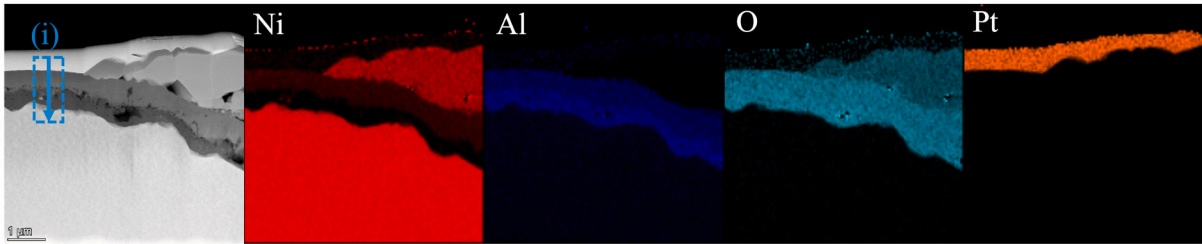
Fig. 3. Relation between the changes in microstructure and the elastoplastic properties obtained from the Ni-Al alloy. (a) Changes in the γ' area fraction and Al concentration obtained from the SEIs and EPMA, respectively, in relation to the average distance from the boundary between the Al depletion zone and the γ/γ' two-phased region. (b) Young's modulus E and (c) hardness H obtained from the inverse analyses of the nanoindentation tests conducted on the Ni-9.8wt.%Al alloy, oxidized at 1100 °C for 1 h. The E calculated from the Al concentration in (a) using JMatPro (blue) are also plotted in (b). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

base superalloy database, plotted in Fig. 3(b) in blue), where the overall results were slightly higher than the experimentally obtained results, though they shared similar trend of increasing with the higher Al concentration. The calculated results are of isotropic Young's modulus for polycrystal Ni-Al alloy. Previous studies show that when considering the anisotropic behavior, (100) plane will show the lowest Young's modulus [57]. Therefore, compared to the experimental results which were obtained from the (100) direction, the actual properties should be slightly lower. In another words, values obtained experimentally in this work are most likely valid, and within reasonable differences. As for H , the results aligned well with the changes in the γ' area fraction and the Al concentration, where significant decrease was observed in the Al depletion zone. Therefore, the average values for the results obtained in the Al depletion zone were selected as the parameters to be incorporated in the FEA model.

Next, the cross-section of the two Ni-Al alloys, (a) low $S_{\text{interface}}$ alloy and (b) high $S_{\text{interface}}$ alloy, both oxidized at 1100 °C for 1 h were observed using STEM-EDS in Fig. 4. The two alloys are the same alloys as the ones used to conduct experimental cross-sectional nanoindentation tests in our previous study [23]. Fig. 4 shows STEM images, EDS maps, and the line profiles of the (i) oxide layers and (ii) the Al_2O_3 /alloy interface. Pt was deposited on the surface of the sample before using FIB to prepare the sample, hence the reason why it was observed in the EDS maps. The results show that the structure of the oxide layers is the same for both samples, as indicated from the 60 at.% of O and 40 at.% of Al measured in the Al_2O_3 layer by the line profiles. EDS maps and line profiles of Al, O, Ni, and S taken from the same sample (figure adapted from [58]) are also shown. Both alloys contain the same bulk S content of 2–3 ppm; however, they exhibited different amounts of S segregation level [58]. The high $S_{\text{interface}}$ alloy showed about 2.0 at.% segregation of S, while only 0.5 at.% of S were observed for the low $S_{\text{interface}}$ alloy. The thickness of the S segregation layer was around 5 nm. Previous research states that amorphous S layers form at the interface [14], which we also assume is the case for this alloy as well. Although it is difficult to prove the uniformity of the S segregation, previous studies observed S segregation in various locations using STEM-EDS, 3D atom probe, and time-of-flight secondary ion mass spectrometry [7,58,59]. All results showed clear segregation of S at the interface, and the alloys prepared using the CaO crucible, as the low $S_{\text{interface}}$ alloy used in this study had been, always showed lower amounts of S segregation level.

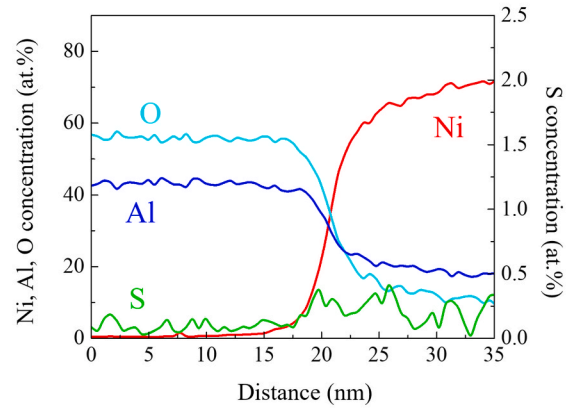
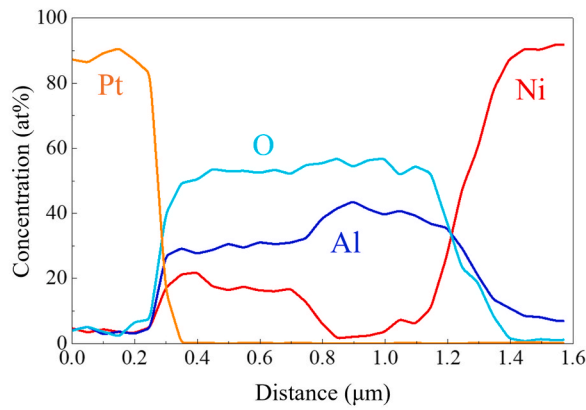
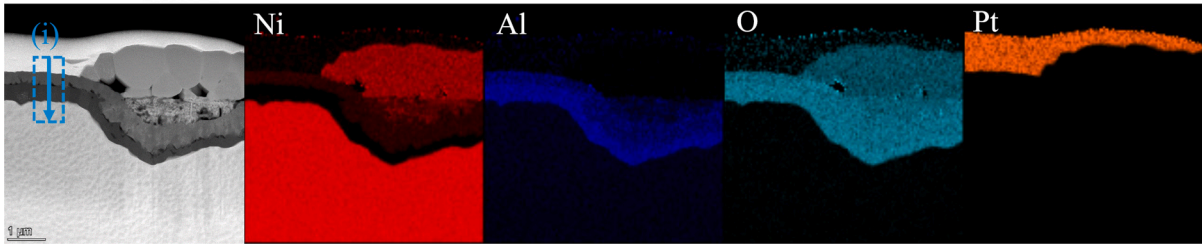
Inverse analyses were conducted on the nanoindentation tests within the Al depletion zone. Fig. 5(a) shows all the load-penetration depth curves obtained from the nanoindentation tests conducted within the Al depletion zone. Inverse analyses were conducted on all of the nanoindentation marks to obtain the E , H , σ_y and B for each result, using the steps shown in section 2.3. The average values were used to create the stress-strain curve in Fig. 5(b), shown as a solid line, while the individual stress-strain curves are shown as dotted lines. The (c-1) AsB image, (c-2) contour map, and (c-3) height profiles of one of the nanoindentation tests are shown as well, taken from the red curve shown in Fig. 5(a).

Fig. 6(a) shows the load-penetration depth curves obtained from the three nanoindentation tests conducted within the grain in the sintered Al_2O_3 . Here, the indentations were conducted using a separate pure Al_2O_3 sample, due to the limited thickness of the actual oxide scale and the difficulty of observing the pile-up within the oxide scale during the inverse estimation. The three results showed relatively similar incline, regardless of the differences in the maximum load. In addition, indentations were clearly observed after the tests, confirming evidence of the plastic deformation of alumina ceramics. Again, inverse analyses were conducted on the nanoindentation marks of these three tests to obtain the E , H , σ_y and B for each result, using the steps shown in section 2.3. Using the average values of the three tests, the stress-strain curve in Fig. 6(b) was obtained by implementing the parameters in eq. (5) and (6), shown in section 2.2. The solid line was drawn using the average parameters, while the dotted lines show the stress-strain curves drawn

(a) Low $S_{\text{interface}}$ alloy

(i)

(ii)

(b) High $S_{\text{interface}}$ alloy

(i)

(ii)

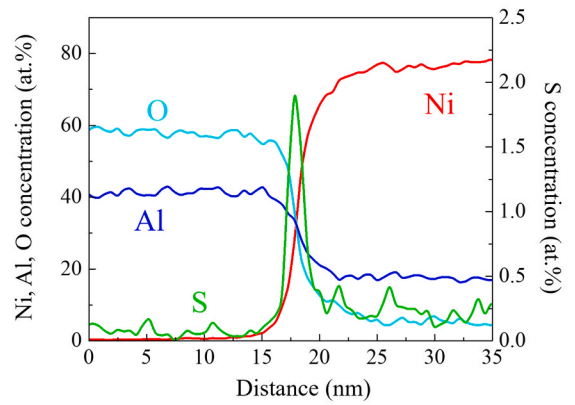
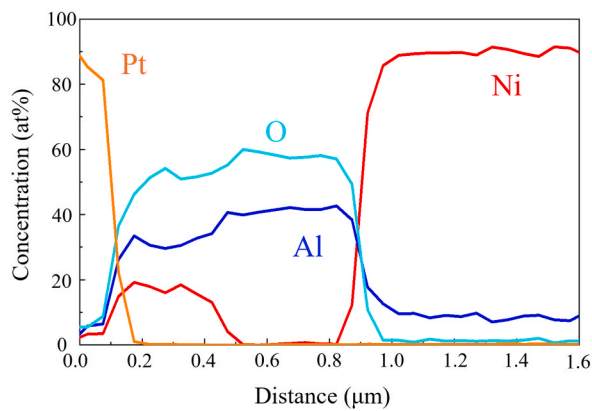


Fig. 4. STEM images, EDS maps and line profiles of (i) Al, O, Ni, and Pt for (a) low $S_{\text{interface}}$ alloy and (b) low $S_{\text{interface}}$ alloy. (ii) Al, O, Ni, and S taken from the same sample (adapted from [58]). The line profiles were taken from the areas indicated in blue. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

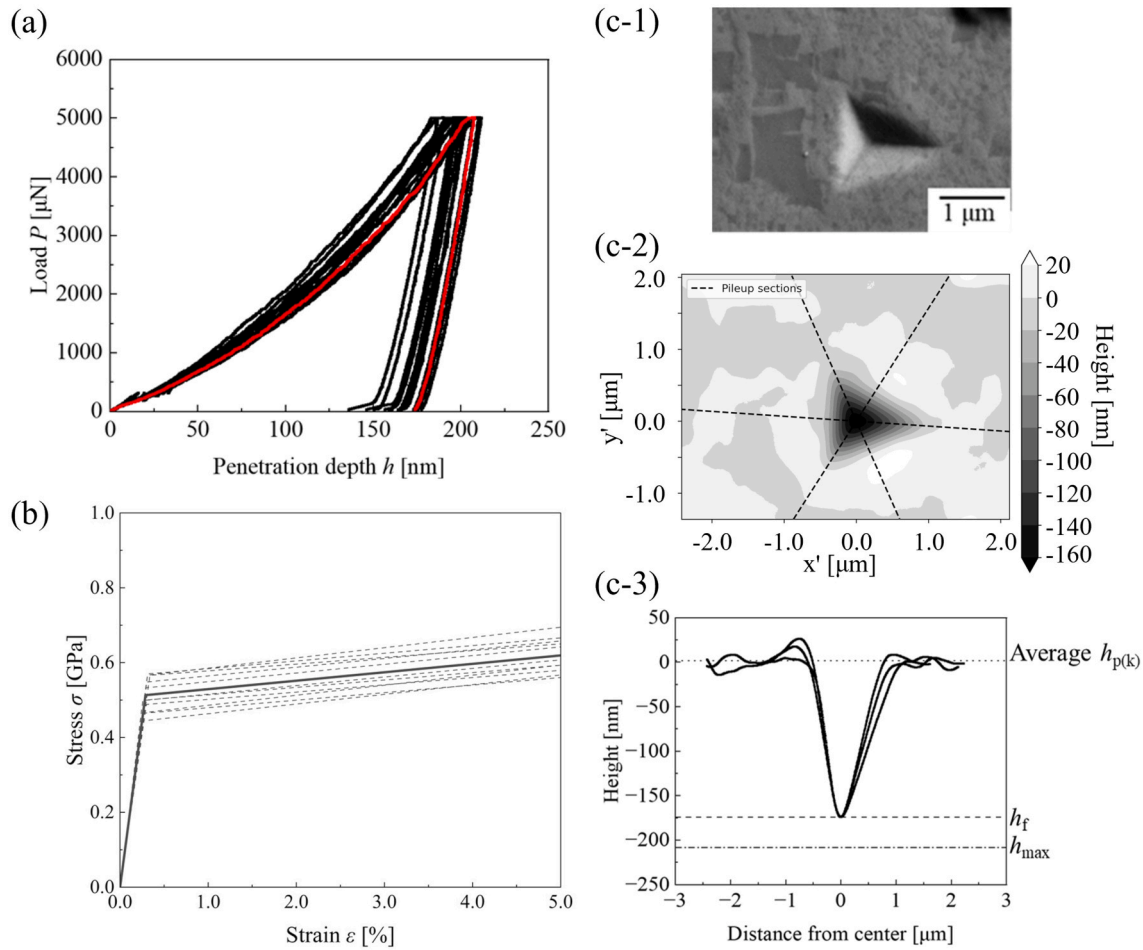


Fig. 5. Results of the inverse analyses of the nanoindentation tests within the Al depletion zone. (a) Load-penetration depth curves for the nanoindentation tests conducted in Al depletion zone of the Ni–Al alloy. (b) Stress–strain curve for Al depletion zone in the oxidized Ni–Al alloy, created according to the linear hardening model, using the parameters obtained from the inverse analysis of the nanoindentation marks. (c-1) AsB image, (c-2) contour map, and (c-3) height profile of one of the nanoindentation marks in the Al depletion zone, corresponding to the red load-penetration depth curve in (a). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

from the individual results. Fig. 6(c-1) shows one of the four AsB images of the nanoindentation marks, which was used to create the contour map in Fig. 6(c-2) by constructing 3-D images of the indentation mark. To differentiate the xyz coordinates from the ones indicated in Fig. 1, the xyz coordinates for the inverse analyses of the nanoindentation marks will be referred to as x' , y' , and z' . In doing so, the height profiles in Fig. 6(c-3) were obtained. These are the results for the load-penetration depth curve shown in red in Fig. 6(a), where the maximum indentation depth $h_{\max}$ and final indentation depth h_f can be determined. The average maximum pile-up height $h_{p(k)}$ (dotted lines) from the 3 lines in Fig. 6(c-2), $h_{\max}$ (alternating long and short dash lines), and h_f (dashed lines) were indicated within the images. The values of E , σ_y and B , along with the standard deviations of the parameters, are shown in Table 2, which were used to plot the stress–strain curves for both the Al_2O_3 sample and the Al depletion zone in the Ni–Al alloy. The Young's moduli for the $\alpha\text{-Al}_2\text{O}_3$ obtained in this study are relatively similar to the values obtained in previous studies [60–62], which incorporate the effects of defects and phase transformations. The yield stress also falls within the range measured in previous study as well [63]. The STEM-EDS results in Fig. 4 also validates that the formed oxide layer is indeed $\alpha\text{-Al}_2\text{O}_3$. Therefore, the parameters used in this study are most likely valid. These stress–strain curves in Fig. 5(b) and Fig. 6(b) will be used to conduct the elastoplastic simulations of nanoindentation close to the interface.

3.2. FEA results for nanoindentation

Fig. 7 shows the comparison of the load-penetration depth curves obtained through nanoindentation [23] (black) and FEA for $d = 0.35$, 0.50 and $0.65 \mu\text{m}$. In addition, cross-sectional nanoindentation test conducted within the Al_2O_3 layer (light blue) [23] and Al depletion zone (gray) are also shown as references, along with the SEIs obtained after testing. Here, the experiment was conducted using the low $S_{\text{interface}}$ alloy, and the actual indentation mark is shown in Fig. 1(b), while the indentation mark for the experimental test conducted within the Al_2O_3 layer is shown in Fig. 7. The P_c value obtained for the test conducted within the Al_2O_3 layer was significantly higher than the P_c values for the tests conducted near the interface [23]. In addition, the slope obtained after the pop-in in the experimental nanoindentation test are closer to the one obtained when tested within the Al depletion zone. These show that the pop-in events observed in these experiments are not due to crack initiations within the Al_2O_3 layer, but are of crack initiation at the interface.

The curves obtained from the FEA are color-coded based on the difference in the initial distance d from the tip of the indenter to the interface: blue for $d = 0.35 \mu\text{m}$, red for $d = 0.50 \mu\text{m}$, and green for $d = 0.65 \mu\text{m}$. This model was selected to assess the reproducibility of the experiment using the elastoplastic finite element model and recreates the curve up to the pop-in event. The decrease in d led to the increase in plastic deformation of the alloy, resulting in the smaller slope of the

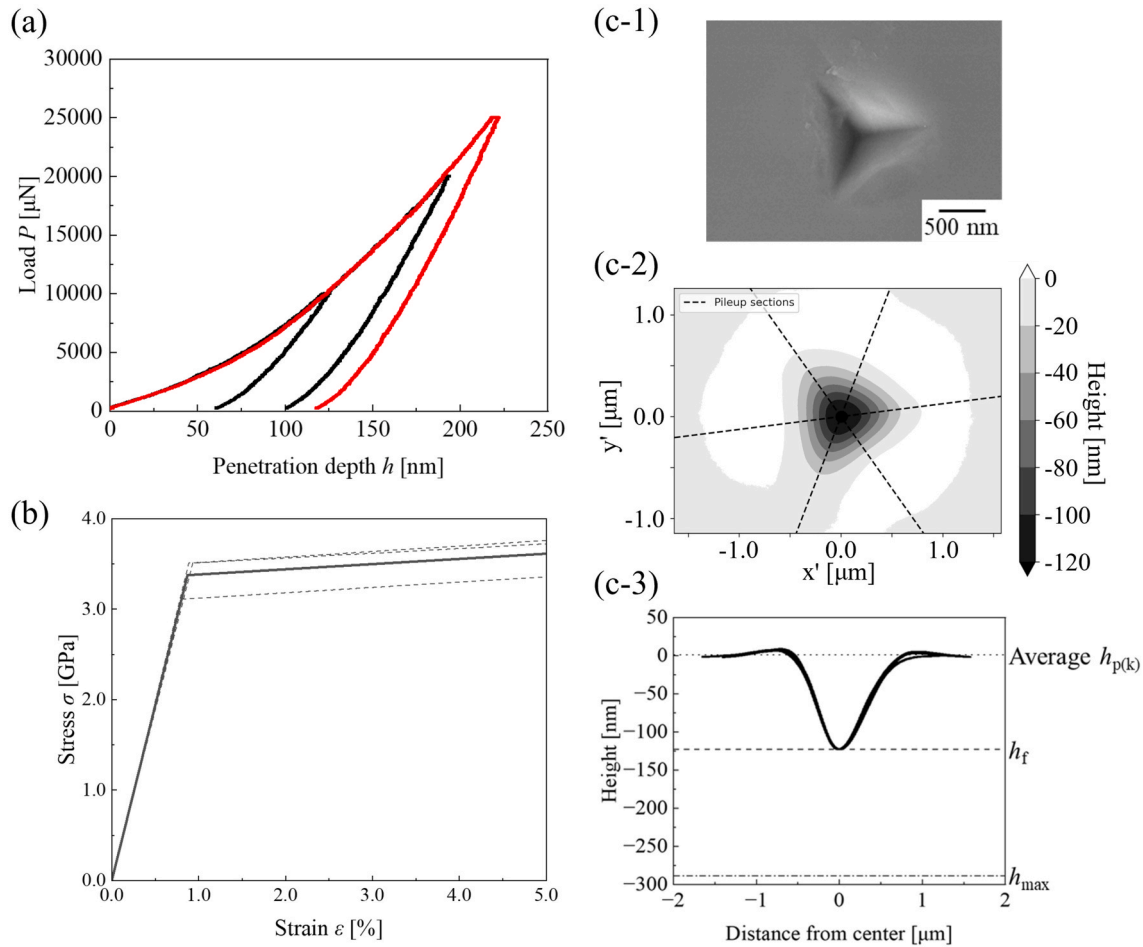


Fig. 6. Results of the inverse analyses of nanoindentation tests within the grain of the sintered Al_2O_3 . (a) Load-penetration depth curves for the nanoindentation tests conducted in the sintered Al_2O_3 sample at the maximum load of 10, 20, and 25 mN, respectively. (b) Stress-strain curve created by the linear hardening model, using the parameters obtained from the inverse analysis of the nanoindentation marks. (c-1) AsB image, (c-2) contour map, and (c-3) height profile of one of the nanoindentation marks, and its corresponding red load-penetration depth curve shown in (a). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Table 2

Parameters used in the FEA, where the values were obtained using nanoindentation tests and inverse analyses.

Material	Material model	Poisson's ratio, ν [-]	Young's modulus, E [GPa]	Yield stress, σ_y [MPa]	Work hardening rate, B [MPa]
Al_2O_3	Bilinear isotropic hardening model	0.24	387.8 ± 8.1	3376.8 ± 188.2	5733.3 ± 370.9
Al depletion zone		0.31	178.2 ± 8.2	513.3 ± 43.5	2250.2 ± 304.1

load-penetration depth curves. However, the results show that overall, the load-penetration depth curve created by the FEA resembles the load-penetration depth curve up to the pop-in event relatively well. This implies that the load-penetration depth curves of the 60-degree indenter on thermally grown oxide scale can be predicted from FEA by using parameters inversely analyzed from curves of 115 degrees-Berkovich indenter on sintered pure Al_2O_3 .

Fig. 8(a-c) and Fig. 9(a-c) show the contour maps of von Mises stress and effective plastic strain around the indenter tip with different distance d , respectively. The indenter is not visualized in these contour maps, and the locations of the interface are pointed out using white arrows. It was confirmed that stress did not extend to the sample edge, indicating the model size was sufficiently large. The points (1–3), indicated in Fig. 6 using white arrows, show the locations where the contour maps were taken. At these points, the indenter reached the penetration depth of (1) $h = 71.8$ nm, (2) $h = 202$ nm, and (3) $h = 314$ nm, respectively. The contour maps show that the smaller d resulted in the amount of plastic strain in the alloy at the same penetration depth to

become larger. It can also be said that the overall trend of the von Mises stress contour maps was relatively similar for all three values of d . At the beginning of the test (point (1)), the stress region takes a circular shape. As the indenter is embedded deeper into the Al_2O_3 scale, the stress region expands, eventually reaching the interface of the two layers. After a while, the interface starts to deform, curving towards the Al depletion zone, and the stress region propagates to the alloy. At point (3) in Figs. 8 and 9 for $h = 314$ nm, the indenter has not reached the interface, as seen from the top and side view of the model shown in Fig. 10. However, the pop-in event observed in the nanoindentation test result in Fig. 7 took place at penetration depth of $h = 314.8$ nm. Therefore, the pop-in event and crack initiation at the interface most likely occurred before the indenter reached the Al_2O_3 /alloy interface.

During the experimental nanoindentation tests, the indentation marks suggested that the indenter will slide towards the Ni-Al alloy [23], where the stiffness of the alloy is significantly lower. To see whether this phenomenon can be observed in the FEA, changes in the x-displacement of the indenter were compared for various conditions. Fig. 11(a) shows

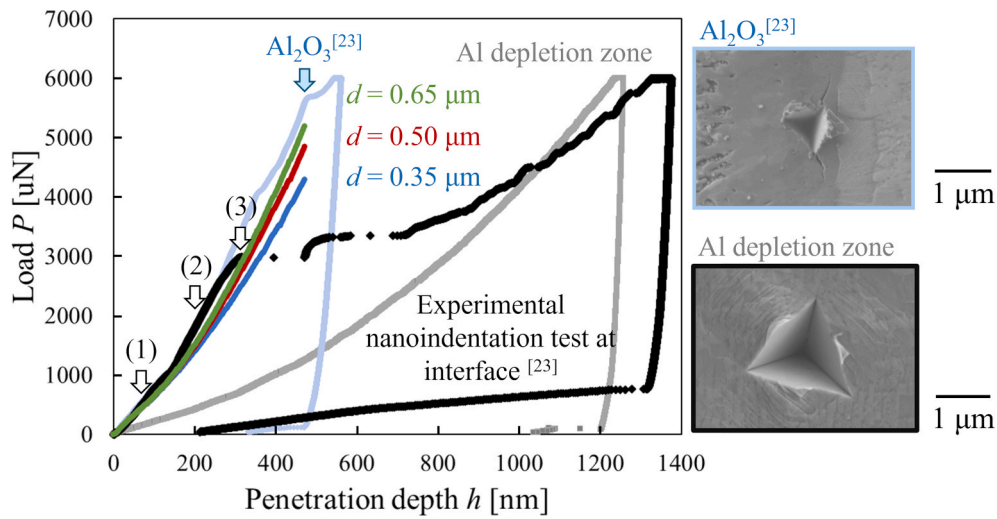


Fig. 7. Load-penetration depth curves obtained through experimental nanoindentation tests [23] (black) and FEA ($d = 0.35 \mu\text{m}$ (blue), $d = 0.50 \mu\text{m}$ (red), $d = 0.65 \mu\text{m}$ (green)), using the parameters from the inverse analyses. The white arrows represent the penetration depths of where the snapshots shown in Figs. 8–12 were taken. The load-penetration depth curves obtained experimentally from the Al_2O_3 layer [23] and Al depletion zone are also shown as a reference, along with SEIs of indentation marks. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

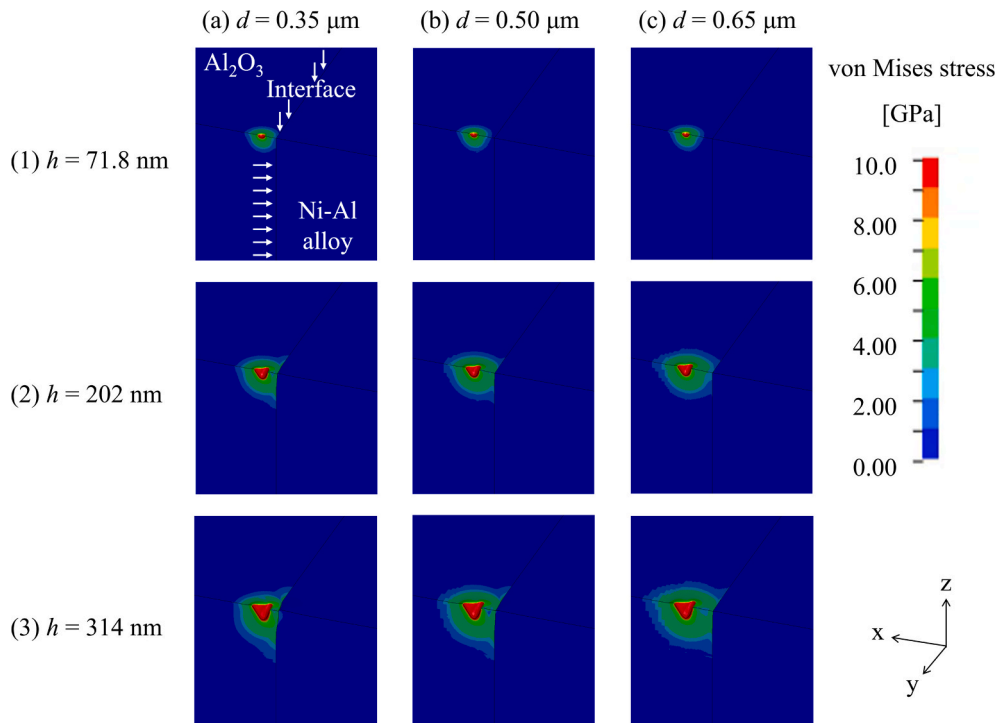


Fig. 8. Changes in the contour maps of von Mises stress at the interface of the Al_2O_3 scale and Al depletion zone in the Ni-Al alloy, in relation to the penetration depth of the indenter during the FEA for (a) $d = 0.35 \mu\text{m}$, (b) $d = 0.50 \mu\text{m}$, and (c) $d = 0.65 \mu\text{m}$. The images represent when the indenter reached the penetration depth of (1) $h = 71.8 \text{ nm}$, (2) $h = 202 \text{ nm}$, and (3) $h = 314 \text{ nm}$.

the snapshots of the movement of the indenter at different times for $d = 0.10 \mu\text{m}$ for (i) $\text{Al}_2\text{O}_3/\text{alloy}$ model and (ii) $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ model. The relation between the x-displacement of the indenter and load P for $\text{Al}_2\text{O}_3/\text{alloy}$ model (white circle) and $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ model (black triangle), are also shown in Fig. 11(b). In addition, the results for $d = 0.35 \mu\text{m}$ (blue), $0.50 \mu\text{m}$ (red), and $0.65 \mu\text{m}$ (green) of the $\text{Al}_2\text{O}_3/\text{alloy}$ model are also shown. Here it is clear that the indenter had moved towards the Ni-Al alloy when trying to indent at the $\text{Al}_2\text{O}_3/\text{alloy}$ interface. On the other hand, no movement in the indenter was observed when conducting indentation at the interface of the $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ model. This shows that the large difference in the parameters such as Young's modulus of Al_2O_3

scale and Ni-Al alloy, shown in Table 2, causes the indenter to slide towards the alloy with lower values of stiffness, in this case the Al depletion zone. Although the FEM in this study does not include a crack propagation model at the interface and therefore cannot simulate pop-in behavior shown in Fig. 7, the FEA result indicates that the evaluation method proposed in this study is useful for evaluating the interfacial strength between alloys and thin oxide surface scales, which have large hardness differences.

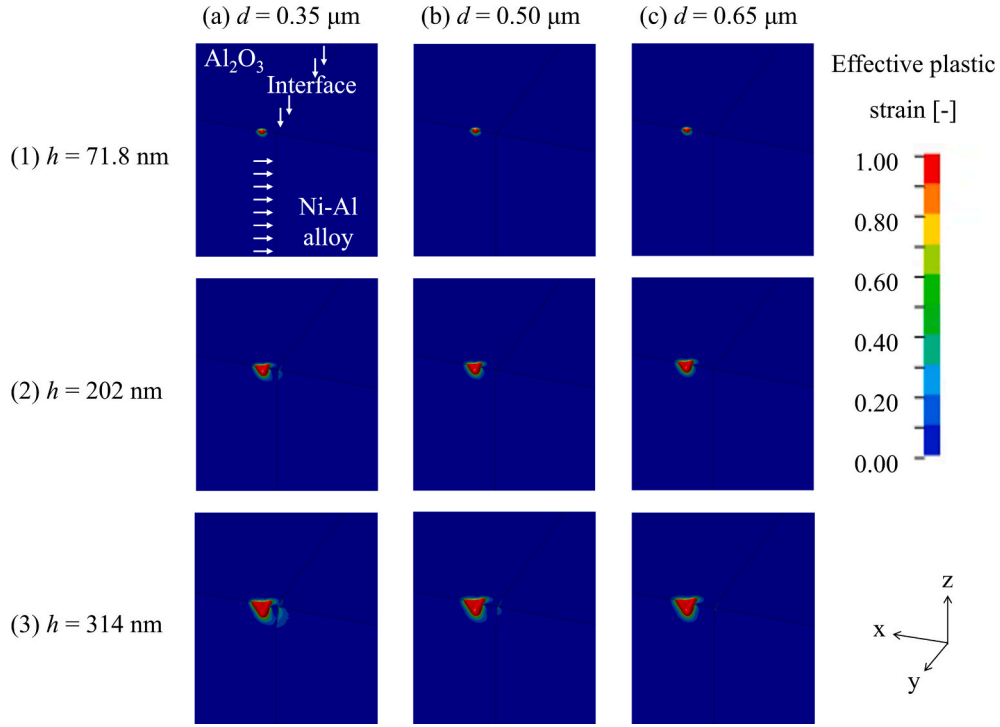


Fig. 9. Changes in the contour maps of effective plastic strain at the interface of the Al_2O_3 scale and Al depletion zone in the Ni-Al alloy, in relation to the penetration depth of the indenter during the FEA for (a) $d = 0.35 \mu\text{m}$, (b) $d = 0.50 \mu\text{m}$, and (c) $d = 0.65 \mu\text{m}$. The images represent when the indenter reached the penetration depth of (1) $h = 71.8 \text{ nm}$, (2) $h = 202 \text{ nm}$, and (3) $h = 314 \text{ nm}$.

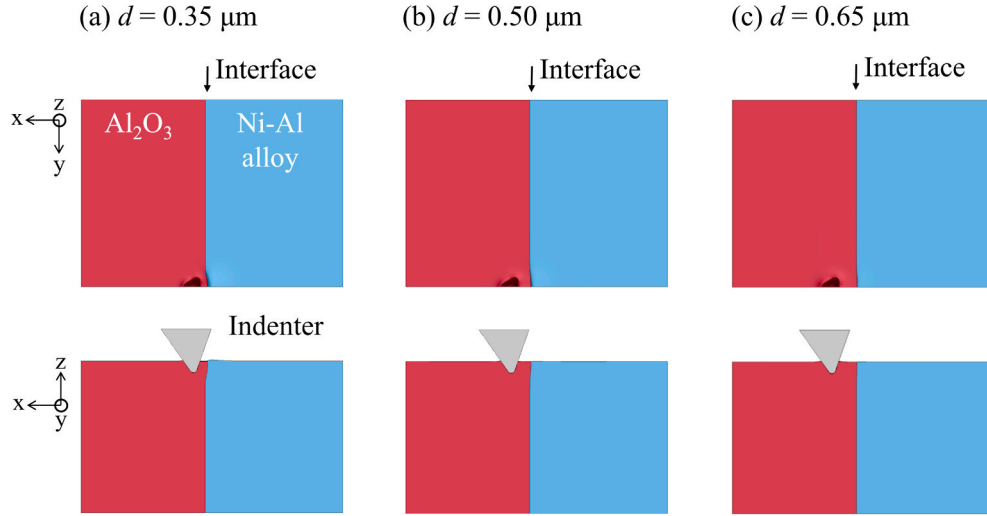


Fig. 10. The indentation mark and the location of indenter in relation to the interface at $h = 314 \text{ nm}$ (point (3) in Fig. 6) for (a) $d = 0.35 \mu\text{m}$, (b) $d = 0.50 \mu\text{m}$, and (c) $d = 0.65 \mu\text{m}$.

3.3. Interfacial stress distribution

Assuming mixed mode fracture (modes I and II), the interfacial stress σ_{Int} was calculated using the following equation:

$$\sigma_{\text{Int}} = \sqrt{\langle \sigma_n \rangle^2 + \sigma_s^2} \quad (7)$$

where σ_n and σ_s are the normal stress and maximum in-plane shear stress that are applied on the interface between the Al_2O_3 scale and Ni-Al alloy, respectively, and $\langle \rangle$ denotes the Macaulay brackets. Figs. 12 and 13 show the contour maps of σ_n and σ_s , respectively, for $d = 0.35, 0.50$, and $0.65 \mu\text{m}$. The images represent when the indenter reached the penetration depth indicated by white arrows in Fig. 7. The images are

the contour maps taken at the interface, near the contact point of the indenter (shows only 1/4 cross-sectional area of the model) and observed from the direction of the alloy to the Al_2O_3 scale. At the beginning, clear increases in both σ_n and σ_s were observed for regions near the indenter tip, regardless of the different distance d . As the indenter is embedded deeper within the Al_2O_3 scale, regions where the maximum σ_s is observed deviate away from the indenter tip. This is indicated by white arrows in Fig. 13, which shows that as the indenter is placed away from the interface, the regions where the maximum σ_s are observed also move away from the indenter tip and closer towards the sides of the indenter.

To understand which parameters affect the crack initiation at the

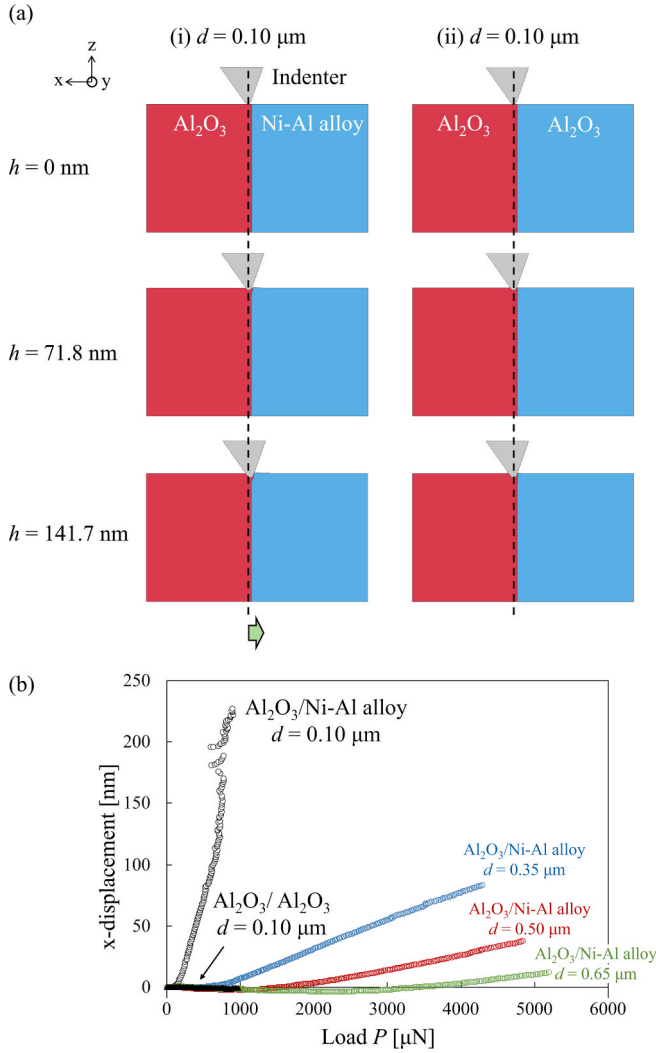


Fig. 11. Changes in the x-displacement of the indenter for nanoindentation tests conducted using various models and initial distance from the interface d . (a) Snapshots of the FEA and model of when the initial distance from the indenter was placed at $d = 0.10 \mu\text{m}$ using (i) Al_2O_3 scale/Ni-Al alloy model and (ii) $\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$ model. (b) Relation between the changes in the x-displacement in relation to the load for various values of d .

interface during testing, the maximum values of σ_n and σ_s out of all elements at the surface of the model creating the interface were compared. These values will be referred to as $\sigma_{n,\max}$ (triangle) and $\sigma_{s,\max}$ (square), respectively, and are plotted in relation to the load P in Fig. 14. The analyses were conducted for all three distances: $d = 0.35$ (blue), 0.50 (red) and 0.65 (green) μm . Here, $\sigma_{n,\max}$ was mostly 0 for all three results, suggesting that $\sigma_{s,\max}$ most likely had a significant effect on the crack initiation at the interface. The maximum value of interfacial stress σ_{Int} out of all nodes at the interface, in relation to the change in load, $\sigma_{\text{Int},\max}$ (circle) are also shown in Fig. 14. This can be obtained by implementing the $\sigma_{n,\max}$ and $\sigma_{s,\max}$ to eq. (7). The changes in $\sigma_{n,\max}$ and $\sigma_{s,\max}$ in relation to the penetration depth h are obtained through FEA, which can be converted to the relation with the load P , as seen in Fig. 7. Therefore, the relation between $\sigma_{\text{Int},\max}$ and P can be expressed. Equation 8 and Table 3 show the equations of approximate curves (white dashed lines) and the coefficient of determination obtained for each region ($P > 1000 \mu\text{N}$).

$$\sigma_{\text{Int},\max} = \sum_{n=0}^6 C_n P^n \quad (8)$$

When the indenter is initially placed closer to the interface, the initial

increase in $\sigma_{s,\max}$ and $\sigma_{\text{Int},\max}$ is faster. Therefore, FEA successfully modeled the nanoindentation experiment, and the relation between $\sigma_{\text{Int},\max}$ and P obtained in this figure correctly depicts the changes in the stresses at the Al_2O_3 /alloy interface, which will be used to convert the P_c to interfacial strength $\sigma_{\text{Int},\max}^c$.

4. Discussion

Interfacial strength $\sigma_{\text{Int},\max}^c$ can be converted from the initial pop-in load P_c , obtained in our previous study [23], by using the approximated equations (eq. (8) and Table 3). Experimental nanoindentation tests were conducted 17 times each for each sample, to compare the P_c values. These values were implemented to eq. (8), which converts the P_c values to $\sigma_{\text{Int},\max}^c$. Fig. 15 (a) and (b) shows the Weibull plots of interfacial strength $\sigma_{\text{Int},\max}^c$ and initial pop-in load P_c [23], respectively, in which the horizontal axes have been converted according to the P values. Again, this simulation was conducted under the assumption that the pop-in event represents the crack initiation at the interface, and that the crack formation at the interface will occur before fracture within Al_2O_3 scale [23]. The dotted lines represent the fitted linear lines obtained by the Weibull plots. The plots represent the average $\sigma_{\text{Int},\max}^c$, which were obtained from the $P-h$ and $\sigma_{\text{Int}}-P$ curves for different d values in Figs. 7 and 14. The error bars represent the minimum and maximum values of $\sigma_{\text{Int},\max}^c$. In Fig. 14, at low values of P (around 1000 μN), the variance in the $\sigma_{\text{Int},\max}$ is large depending on the value of d . Therefore, the variance in the calculated $\sigma_{\text{Int},\max}^c$ values were significantly large for P_c values with low fracture probability F .

The values obtained from the Weibull distribution in Fig. 15(a) are shown in Table 4. The interfacial strengths for both samples were followed by Weibull distribution, which is based on the weakest link model, similar to P_c . This means that the initiation of interface cracks, similar to fracture in brittle ceramics, is likely due to the existence probability of structural defects acting as the weakest link. Meanwhile, both the scale parameter β and Weibull modulus m for the alloy with high amount of interfacial S segregation (high $S_{\text{interface}}$ alloy) were smaller than that for the alloy with low amounts of segregated S (low $S_{\text{interface}}$ alloy), similar to P_c . However, the ranking of the $\sigma_{\text{Int},\max}^c$ values differ from the ranking of the P_c values obtained from the previous experiment, which also affects the β and m values. The results show that, although the quantitative data may vary with additional tests, the β had increased from 2.64 GPa to 2.79 GPa by the suppression of local interfacial S segregation level [58]. Meanwhile, clearer differences appear within low fracture probability regions. Although it is difficult to precisely compare the data quantitatively due to the limited number of data points, the fracture probability $F = 4\%$, the largest differences in the $\sigma_{\text{Int},\max}^c$ values of approximately 780 MPa between high $S_{\text{interface}}$ alloy ($\sigma_{\text{Int},\max}^c = 1.73$ GPa) and low $S_{\text{interface}}$ alloy ($\sigma_{\text{Int},\max}^c = 2.51$ GPa) can be seen in the Weibull distribution. Since the delamination of the protective oxide scale during the cyclic oxidation of turbine blade could start at the weakest interface between Al_2O_3 scale and the Ni-Al alloy, the difference in interfacial strength of 780 MPa may have a significant impact on cyclic oxidation property. Further, it is interesting to note that the interfacial strength remains constant regardless of the S content within high F region, while it varies according to the weakest link model [64] and decreases with increasing S content within low F region. In previous study, S segregation level on interface was evaluated by STEM-EDS technique and local values [7]. The change in the S amount due to the interface structure according to the grain orientation of Al_2O_3 or due to the position could not be evaluated. This suggests the following: (a) a maximum value for the amount of S segregation may exist depending on the grain boundary structure in the high F region, resulting in the interfacial S levels being roughly equivalent, or that; (b) a grain boundary structure existed in which the grain boundary strength was insensitive to the amount of S despite the interfacial S levels being

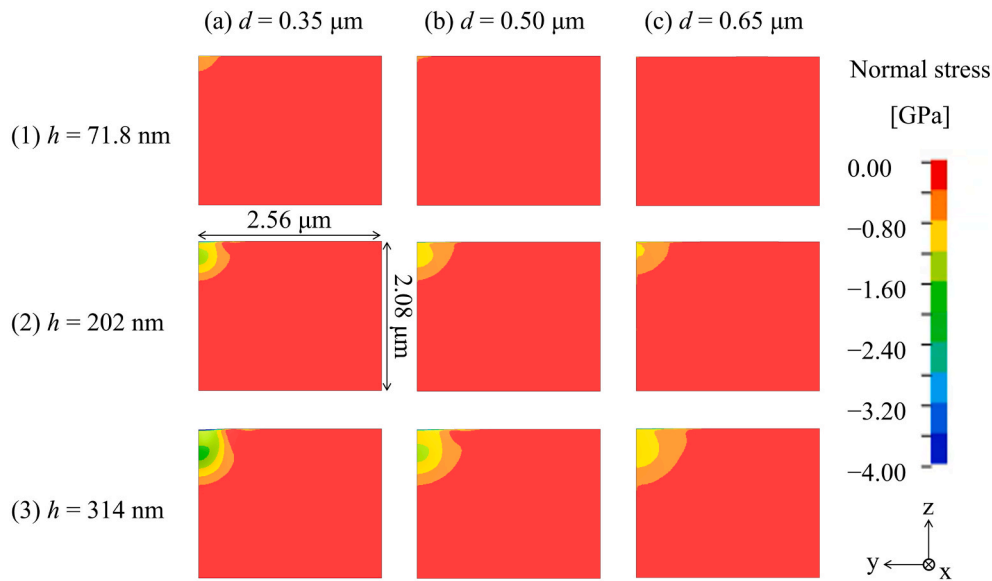


Fig. 12. Changes in the contour maps of normal stress σ_n at the interface of the Al_2O_3 scale and Al depletion zone in the Ni-Al alloy, in relation to the penetration depth of the indenter during the FEA for (a) $d = 0.35 \mu\text{m}$, (b) $d = 0.50 \mu\text{m}$, and (c) $d = 0.65 \mu\text{m}$. The images represent when the indenter reached the penetration depth of (1) $h = 71.8 \text{ nm}$, (2) $h = 202 \text{ nm}$, and (3) $h = 314 \text{ nm}$. The regions shown are 1/4 cross-sectional area of the model, focusing on the region near the contact point of the indenter tip.

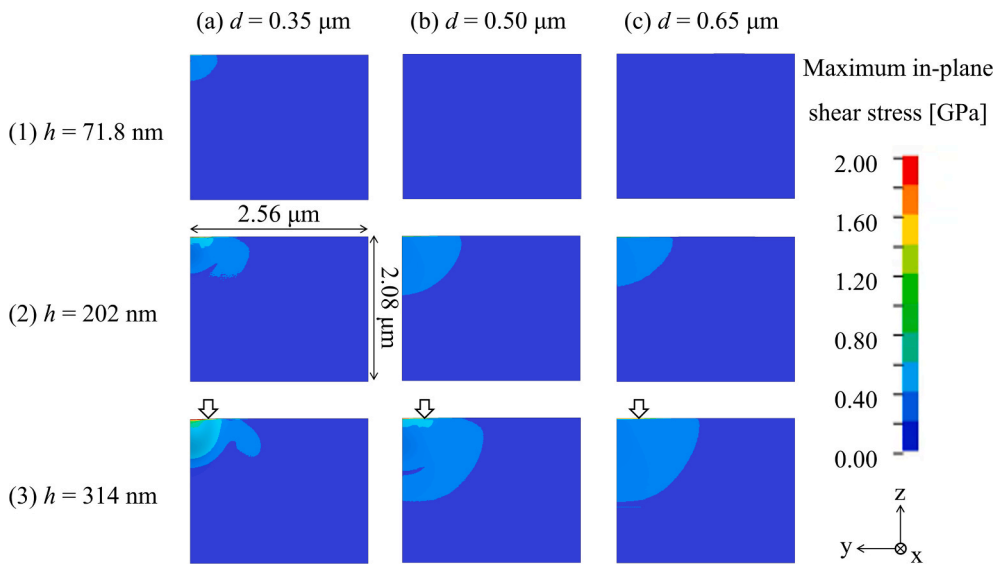


Fig. 13. Changes in the contour maps of maximum shear stress σ_s at the interface of the Al_2O_3 scale and Al depletion zone in the Ni-Al alloy, in relation to the penetration depth of the indenter during the FEA for (a) $d = 0.35 \mu\text{m}$, (b) $d = 0.50 \mu\text{m}$, and (c) $d = 0.65 \mu\text{m}$. The images represent when the indenter reached the penetration depth of (1) $h = 71.8 \text{ nm}$, (2) $h = 202 \text{ nm}$, and (3) $h = 314 \text{ nm}$. The regions shown are 1/4 cross-sectional area of the model, focusing on the region near the contact point of the indenter tip. The white arrows represent where the maximum σ_s were detected.

different.

For this research, the shear stress was dominant for crack initiation at the interface. However, for material design at an industrial level, criterion for normal stress is also necessary. There are two likely methods to determine this criterion: (1) conduct experiments similar to this research where normal stress is the dominant factor, or (2) assume the ratio between the shear strength (mode II fracture stress) and normal strength (mode I fracture stress). If the materials are the same, regardless of the changes in stress within the sample, the same interfacial strength obtained in this study can be used for strength evaluation. In addition to interfacial strength, obtaining the fracture energy is also crucial for clarifying the crack propagation phenomena at the interface. To do so, additional tests and analyses for conventional Ni-base superalloys using

a cohesive-zone model is most likely effective, which will be for future work.

5. Conclusion

To conclude, the following has been made clear about the method of using inverse analyses of nanoindentation tests and FEA of the cross-sectional nanoindentation technique to assess the Al_2O_3 scale/Ni-Al alloy interfacial stress:

1. Inverse analyses of the nanoindentation tests conducted using the Berkovich indenter on the different regions of the Ni-Al alloy and the sintered Al_2O_3 sample were proven useful in obtaining the

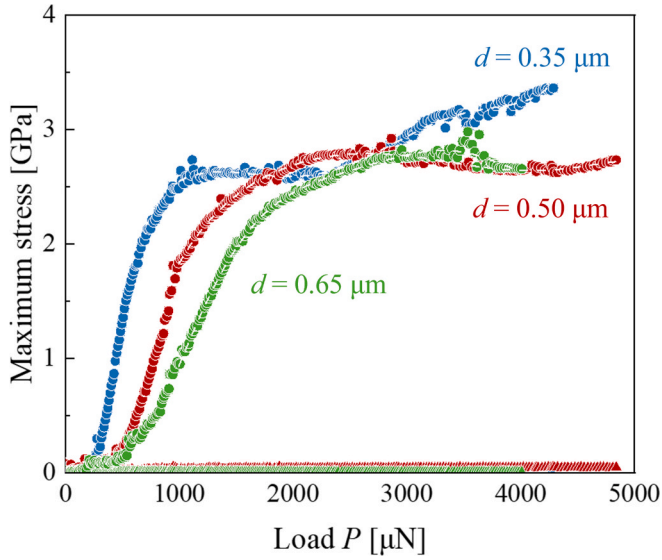


Fig. 14. Relation between the maximum values of $\sigma_{n, \max}$ (triangle), $\sigma_{s, \max}$ (square), and $\sigma_{\text{Int}, \max}$ (circle), obtained from the elements at the surface of the model creating the interface between the Al_2O_3 scale and Ni-Al alloy. The white dashed lines represent the approximated curves for the relation between P and $\sigma_{\text{Int}, \max}$.

Table 3

Parameters for the approximate curve (equation 8) obtained for each distance d and the coefficient of determination.

d [μm]	$C_6 \times 10^{-20} \left[\frac{\text{GPa}}{(\mu\text{N})^6} \right]$	$C_5 \times 10^{-16} \left[\frac{\text{GPa}}{(\mu\text{N})^5} \right]$	$C_4 \times 10^{-12} \left[\frac{\text{GPa}}{(\mu\text{N})^4} \right]$	$C_3 \times 10^{-8} \left[\frac{\text{GPa}}{(\mu\text{N})^3} \right]$	$C_2 \times 10^{-6} \left[\frac{\text{GPa}}{(\mu\text{N})^2} \right]$	$C_1 \times 10^{-3} \left[\frac{\text{GPa}}{\mu\text{N}} \right]$	C_0 [GPa]	R^2
0.35	1.96	-2.45	1.08	-0.179	0.0716	2.54	0.874	0.980
0.50	-0.624	1.11	-0.792	0.301	-6.71	8.77	-2.55	0.988
0.65	5.31	-7.97	4.72	-1.38	20.2	-11.7	2.29	0.995

elastoplastic properties of the materials. Clear changes in the γ' area fraction were observed by the depletion in the Al content, caused by the oxidation of the alloy, leading to the decrease in the Young's modulus and hardness of the alloy. The stress-strain curves were created using the parameters obtained from the inverse analyses. These values were used in the FEA to model the nanoindentation tests for the evaluation of the Al_2O_3 /alloy interface.

- FEA successfully modeled the nanoindentation test conducted in our previous study by recreating the load-penetration depth curve up to the pop-in event. From the model it was clear that the pop-in events likely occurred before the indenter itself reached the interface. Indentation near the interface showed that due to the large difference in the parameters such as Young's modulus of Al_2O_3 and Ni-Al alloy, the indenter will slide towards the alloy with lower values of stiffness.
- The normal stress σ_n and maximum shear stress σ_s at the interface were obtained from the FEA. The values of $\sigma_{\text{Int}} (= \sqrt{\langle \sigma_n \rangle^2 + \langle \sigma_s \rangle^2})$ were used to compare the interfacial stress between alloys with different amounts of interfacial S segregation. Maximum shear stress σ_s had a significant effect on the crack initiation at the interface for this research. The relation between maximum interfacial stress out of all the elements at surface of the model creating the interface, $\sigma_{\text{Int}, \max}$, and the load P correctly depicted the changes in the stresses at the Al_2O_3 /alloy interface. This was used to convert the Weibull distribution for the initial pop-in loads P_c to interfacial strengths $\sigma_{\text{Int}, \max}^c$.

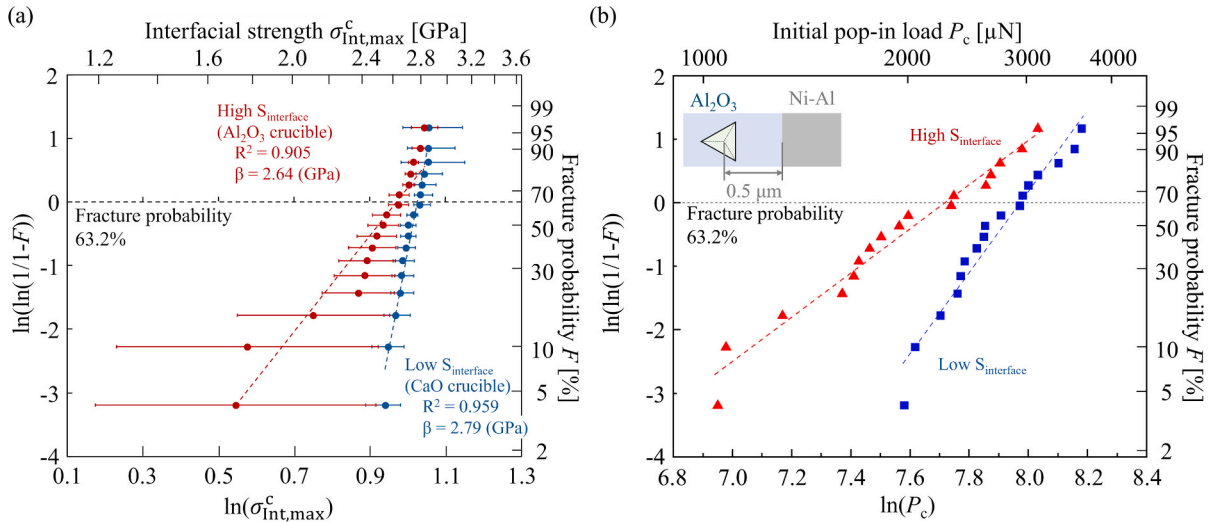


Fig. 15. Weibull plots of (a) interfacial strength $\sigma_{\text{Int}, \max}^c$ for high $S_{\text{interface}}$ (alloy with higher amounts of S segregation at interface) and low $S_{\text{interface}}$ alloys (lower amounts of S segregation). The values were converted from (b) initial pop-in load P_c (adapted from [23], with permission from SpringerNature).

Table 4

Parameters and values obtained from the Weibull distribution in Fig. 12(a).

Material	S segregation level (STEM-EDS, [at.%])	m [-]	β [GPa]	$\sigma_{\text{Int}, \max}^c$ when $F = 96\%$ [GPa]	$\sigma_{\text{Int}, \max}^c$ when $F = 4\%$ [GPa]
Low $S_{\text{interface}}$	0.5	30.8	2.79	2.90	2.51
High $S_{\text{interface}}$	2.0	7.54	2.64	3.08	1.73

The interfacial strength varies according to the weakest link model; at fracture probability $F = 4\%$, the largest differences in the $\sigma_{\text{int,max}}^c$ values of approximately 780 MPa between high $S_{\text{interface}}$ alloy ($\sigma_{\text{int,max}}^c = 1.73$ GPa) and low $S_{\text{interface}}$ alloy ($\sigma_{\text{int,max}}^c = 2.51$ GPa) were seen in the Weibull distribution.

CRedit authorship contribution statement

Chihiro Tabata: Writing – review & editing, Writing – original draft, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Taiyo Maeda:** Writing – review & editing, Software, Resources, Investigation, Formal analysis, Data curation. **Thomas Hoefler:** Writing – review & editing, Methodology, Data curation. **Shingo Ozaki:** Writing – review & editing, Software, Resources, Formal analysis. **Kyoko Kawagishi:** Writing – review & editing, Supervision. **Shinsuke Suzuki:** Writing – review & editing, Supervision. **Takahito Ohmura:** Writing – review & editing, Software, Resources, Methodology. **Toshio Osada:** Writing – review & editing, Validation, Supervision, Resources, Methodology.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Chihiro Tabata reports financial support was provided by Japan Society for the Promotion of Science. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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