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Combined Effect of Cold Working and Al Content on Oxidation Behavior of Ni-Base Alloys at 900 °C and 1000 °C



WOJCIECH J. NOWAK, ANTON CHYRKIN, and TIMUR GALIULLIN

Ni-base alloys are the most commonly used type of construction materials, especially in high-temperature applications. It is widely known that the high-temperature corrosion resistance of these types of materials depends on their chemical composition. Specifically, the Cr and Al content plays an important role. The formation of an alumina scale (aluminum oxide) is the most desired phenomenon because it provides the best protection against high-temperature oxidation among all known types of oxide scale. The effect of the Al content of Ni-base alloys on the transition from internal to external oxide scale formation has been widely investigated. It is also known that cold working can sufficiently affect the oxidation kinetics. However, there is a lack of knowledge about the synergetic effect of the Al content and cold working on the transition from internal to external scaling. Therefore, the objective of the present study is to explore the interaction between the Al content of the NiCrAl base alloy and its surface finish to evaluate the extent of a positive effect of cold working that could compensate for an insufficient Al content in the Al alloy from external Al₂O₃. To elucidate this effect, three commercially available Ni-base superalloys, namely IN 617, IN 602 CA, and IN 692 with different amounts of Al and different cold-worked surfaces, were investigated during air oxidation at 900 °C and 1000 °C up to 48 h. The results showed that cold grinding pronounced the formation of alumina scale in the case of alloys with insufficient Al content. The reasons for such an effect have been elucidated and are explained.

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I. INTRODUCTION

WROUGHT Ni-base alloys are state-of-the-art construction materials for various high-temperature applications requiring high creep strength as well as oxidation resistance. The common industrial applications for these alloys are in gas burners, furnace rollers, heat exchangers, flame tubes, etc. Alloy grades based on the Ni–Cr system such as IN $617^{[1,2]}$ and IN $625^{[3,4]}$ rely for their corrosion resistance on the formation of a protective, well-adherent, slow-growing Cr_2O_3 scale.

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Alloys developed based on the Ni–Cr–Al system such as 602 CA,^[5] HR-214,^[6] and HR-224^[7] can operate at higher temperature as they are designed to grow an external α -Al₂O₃ scale on the surface of the components. Depending on the exposure conditions (temperature, exposure time, atmosphere, and thermal cycling), these alumina-forming alloys are sometimes incapable of forming a protective, external alumina scale.^[8–10] The transition from protective to nonprotective oxidation (and vice versa) in commercial Ni-base alloys is an extremely important phenomenon for the practical use of these materials and further alloy optimization.

The oxidation behavior of NiČrAl-base alloys was fundamentally studied by Giggins and Pettit,^[11] who mapped the oxidation morphology as a function of the Cr and Al content. Alloys with low Cr and Al contents are prone to form external NiO along with internal oxidation of Cr and Al (type I morphology). High-Cr alloys with low Al content below 2.5 wt% form external chromia scales followed by an internal oxidation zone (IOZ) of Al₂O₃ (type II).^[10,12–14] Depending on temperature, an alumina-forming alloy (type III) requires at least 15 and 3 wt% Cr. Further alloying of the system with Ta, Ta W, etc. may somewhat shift the boundary between types II and I;^[15] however, the main factor

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promoting Al_2O_3 scaling is the Al content, which often cannot be increased for metallurgical or technical reasons such as weldability, machinability, suitability for additive manufacturing, etc.

Another important factor affecting the oxidation morphology of NiCrAl-base alloys is the surface finish. Cold-work produced by grinding, blasting, or shot-peening is well known to promote a protective oxidation behavior of high-temperature alloys.[16-21] Deformation of the near-surface layer injects numerous defects into the alloy subsurface, which leads to recrystallization and provides short-circuit diffusion paths for Al and Cr, thus enhancing their transport toward the surface. A positive effect of cold work of surfaces was also found for stainless steels exposed in O_2 ,^[22] air^[23] and even in ultra-supercritical steam.^[24] The grain size effect has also been investigated for Ni-base superalloys. Grain refinement in GTD111 was found to increase the oxidation resistance.^[25] Similar observations were made for IN740H.^[26] In contrast, grain refinement by additive manufacturing of IN625 was found to have no effect on the oxidation resistance in air, while the application of heat treatment for grain coarsening was positive.^[27] Thus, the observations seem to be somewhat contradictory. The aim of the present study is to explore the interplay between the Al content of NiCrAl-base alloy and its surface finish to evaluate the extent of a positive effect of cold work, potentially compensating for an insufficient Al content in the alloy to from external Al_2O_3 .

II. EXPERIMENTAL

In the present work, three commercially available Ni-base superalloys were investigated. The nominal chemical composition of the alloys is given in Table I. Rectangular specimens measuring $20 \times 10 \times 2 \text{ mm}^3$ were machined from as-cast alloy ingots. Surfaces of specimens were prepared in up to three different stages: (i) polished to 1 μ m with diamond paste, and ground using SiC paper with (ii) 1000 and (iii) 80 grit to obtain different surface roughness. The surface roughness was measured using a contact profilometer (Hommel Werke T8000), where the traverse length was 4 mm while the linear speed was -0.5 mm/s. For each sample, five measurements of surface roughness were performed. The measurement direction was set as the direction perpendicular to the directions of scratches on the surface of the samples, if applicable. Prior high-temperature exposure samples were ultrasonically cleaned in acetone, dried with pressurized air, and subjected to isothermal oxidation test at 900 °C and 1000 °C for 48 h in a tube furnace (Xerion X-Tube) in air. The air flow was fixed at 2 L/min. Before and after exposure, the weight of the samples was measured by using a microbalance RADWAG WAA 100/C/1 with 0.1 mg accuracy to determine the oxygen uptake. To ensure the reproducibility of the obtained results, two specimens per condition were exposed. The results obtained showed deviations of less than 5%, thus error bars are not shown on the plots. After oxidation tests, the samples were examined by scanning electron microscopy (SEM) to analyze the surface morphology. After surface observation, selected samples were investigated by glow discharge optical emission spectrometry (GD-OES) in terms of the element concentration distribution in the oxide scale and also in the bulk alloy. The obtained results showed the intensities of light emitted by atoms of certain elements as a function of sputtering time. These profiling results were requantified using so-called relative sensitivity factors (RSFs) as described elsewhere.^[28–30] After GD-OES analyses, the microstructure and chemical composition of the oxide scales were



Fig. 1—Roughness profiles obtained on surfaces: (a) polished (1 μ m), (b) ground using 1000 grit, and (c) ground using 80 grit sandpapers.

observed on cross-sections using SEM. To reveal grains by etching of grain boundaries, cross-sectioned samples



Fig. 2—Area-specific mass change after 48 h for alloys IN 617, 602 CA, and IN 693 during air oxidation at 900 $^\circ$ C and 1000 $^\circ$ C.



Fig. 3—Oxidation rate k'_w after 48 h for alloys IN 617, 602 CA, and IN 693 during air oxidation at 900 °C and 1000 °C (Color figure online).

were electrochemically etched using 10% oxalic acid solution. Image analyses were performed at five locations on the samples cross-sections using NIS-Elements 2.3 software.

III. RESULTS

A. Surface Roughness

Figure 1 shows a typical roughness measurement obtained on the polished surface (1 μ m; Figure 1(a)), ground surface (1000 grit; Figure 1(b)), and 80 grit (Figure 1(c)). The roughness parameters are summarized in Table II. The lowest roughness parameters were obtained for polished surfaces, intermediate values for the medium ground ones (1000 grit), and the highest ones for surfaces ground using 80 grit sandpaper. The R_a parameter varies within one order of magnitude as a result of surface finish, while the R_q , R_z , and S_{DR} parameters are similar for the 1 μ m and 1000 grit

sandpaper, being higher only for the surface ground to 80 grit.

B. Oxidation Kinetics

The oxygen uptake results obtained for the three alloys studied with three different surface finishes are shown in Figure 2. The mass change values obtained for all the studied alloys, irrespective of the surface finish, were higher after oxidation at 1000 °C compared with 900 °C. A clear trend can be observed at both studied temperatures. For IN 617, a mass change increase can be found for specimens with higher surface roughness. At the same time, an increased surface roughness results in lower oxygen uptakes for both alloy 602CA and IN 693 at 900 and 1000 °C. The most vivid effect of surface finish was observed for IN 693 when exposed at 1000 °C (dashed green curve in Figure 2). A similar, albeit less pronounced, trend was observed for 602 CA at 900 °C (solid blue line). On the basis of the obtained mass change, the oxidation rate was calculated and is plotted in Figure 3. Since further analyses showed formation of chromia and/or alumina oxide scale, the oxidation rate was calculated under the assumption of a parabolic rate law. In general, similar observations were made as for the basis of the mass change plot, namely the highest oxidation rate at both investigated temperatures was found for IN 617, and the lowest for alloy 693 (Figure 3).

C. Oxidation at 900 $^{\circ}C$

1. Morphology of oxide scales

Figure 4 show SEM/backscattered electron (BSE) top-view images of the oxidized alloy surfaces after air exposure for 48 h at 900 °C. For IN 617, eternal chromia scale was observed after all three types of surface preparation. Remarkably, the oxide scale grown on the polished and 1000 grit ground surfaces showed characteristic protrusions at alloy grain boundaries (GBs; Figure 4(a) and (b)). Such an effect is not visible on the oxide scale grown on 80 grit ground IN 617 (Figure 4(c)). For IN 602 CA, the alloy surface of the polished and 1000 grit ground specimens was covered with continuous and uniform chromia scale (Figures 4(d) and (e)), while the 80 ground 602 CA specimen demonstrated large areas covered by external alumina. For IN 693, uniform chromia scale was observed only on the polished surface (Figure 4(g)). The 1000 grit ground surface showed wide regions covered with alumina together with islands of Cr₂O₃ nodules (Figure 4(h)). The 80 grit ground surface was nearly fully covered with alumina, with only a few nodules of chromia scale visible (Figure 4(i)).

2. GD-OES depth profiles

To investigate the distribution of the elements within the oxide scale and in the base alloy, GD-OES depth profiles were measured on the studied alloy specimens with polished and 80 grit ground surfaces. The obtained results are shown in Figure 5. The GD-OES profiles showed that Cr is strongly enriched in the outer part of



Fig. 4—SEM/BSE images of the surfaces of IN 617 (*a* through *c*), 602 CA (*d* through *f*), and IN 693 (*g* through *i*) after 48 h of air oxidation at 900 $^{\circ}$ C with: polished surface (a, d, g), 1000 grit (b, e, h), and 80 grit (c, f, i).

Table I. Nominal Chemical Composition of Studied Alloys

Name of the Alloy	Element Content (wt%)											
	Ni	Cr	Co	Мо	Nb	Al	Ti	Fe	Mn	Si	Cu	С
IN 617 Alloy 602 CA	BASE BASE	22.0 25.0	12.5	9.0		1.2 2.1	0.6	3.0 9.5	0.5	0.5	0.5	0.1
Alloy 693	BASE	29.0			1.5	3.3	0.5	4.3	0.5	0.3	0.3	0.2

the oxide scale of IN 617 for both surface preparations (Figures 5(a) and (b)). Slight enrichment of Ti was also observed at the gas-oxide interface. The oxide scale was followed by an increase of Al, but the time/depth of this Al enrichment was different for the polished and ground surfaces, i.e., shallower for the polished specimen (Figure 5(a)) as compared with the ground (Figure 5(b)) surface. For alloys 602 (Figures 5(c) and (d)) and 693 (Figures 5(e) and (f)), the influence of the surface preparation method was clearly observable. For the polished surfaces, the outer scale was enriched in the Cr profile, while the ground specimens demonstrated

enrichment of Al (see Figure 5(d) and especially Figure 5(f)). None of the obtained GD-OES profiles showed any significant increase in the Fe profile (but with a slight enrichment in Fe content (below 1 at%) in the outer part of the oxide scale in IN 617 (Figure 5(a) and (b)) despite the relatively large difference in the Fe content among the alloys in the as-received condition. This implies that Fe does not actively participate in the oxidation process and its influence can be neglected here.



Fig. 5—GD-OES depth profiles of IN 617 (a, b), 602 CA (c, d), and IN 693 (e, f) after 48 h of air oxidation at 900 °C: polished (a, c, e) and 80 grit ground (b, d, f) surface.

3. Cross-sections

Metallographic cross-sections of IN 617 specimens after air oxidation at 900 °C for 48 h revealed a classical type II (according to Giggins and Pettit^[11]) oxide scale morphology consisting of an external Cr_2O_3 oxide scale and internal precipitates of Al_2O_3 (Figures. 6(a) through (c) and 7(a) and (b)). Such morphologies were observed after all three surface preparation methods. Cross-sections of 602 CA of the polished and 1000 grit ground surfaces revealed a similar morphology, i.e., an outer chromia scale followed by internal oxidation of Al, but the thickness of the outer chromia scale was lower as compared with that for IN 617 (Figures 6(d) and (e) and 7(c)). In the case of the 80 grit ground 602 CA specimen, an outer chromia scale was followed by a virtually continuous sublayer of Al_2O_3 (Figure 6(f)), and locally only external alumina scale with local chromia nodules was formed (Figure 7(d)). The polished IN 693 specimen



Fig. 6—SEM/BSE images of the cross-sectioned IN 617 (*a* through *c*), 602 CA (*d* through *f*), and IN 693 (*g* through *i*) after 48 h of air oxidation at 900 °C: polished surface (a, d, g), 1000 grit ground surface (b, e, h), and 80 grit ground surface (c, f, i).

	surfaces						
	Surface preparation						
Roughness parameter	1 µm	1000 grit	80 grit				
$\overline{R_{\rm a}~(\mu {\rm m})}$	0.0095	0.0113	0.4731				
$R_{\rm q}$ ($\mu {\rm m}$)	0.0138	0.0151	0.6282				
$R_{\rm z}(\mu{\rm m})$	0.0636	0.0846	3.9126				
S _{DR} (%)	0.0004	0.0009	3.2315				

 Table II.
 Calculated roughness parameters for studied surfaces

formed a duplex oxide scale consisting of an outer chromia layer and an internal oxidation zone of Al_2O_3 precipitates (Figures 6(g) and 7(e)). The same alloy after 1000 grit treatment formed a nearly continuous alumina sublayer (Figure 6(h)) beneath the external Cr_2O_3 . Finally, the 80 grit specimen was able to grow an external and continuous alumina accompanied by local external nodules of Cr-rich oxide (Figures 6(i) and 7(f)).

D. Oxidation at 1000 $^{\circ}C$

1. Morphology of formed oxide scale

Air oxidation at 1000 °C generally revealed a similar trend as observed for the alloys exposed at 900 °C. However, minor changes were also detected. In the case of IN 617, the presence of oxide ridges at grain boundaries (GBs) was observed on the polished and 1000 grit ground surface (Figure 8(a) through (c)) but was absent on the 80 grit ground specimen. The surface of 602 CA was mostly covered by chromia scale (Figure 8(d) through (f)). Only on the 80 grit surface of 602 CA was a local island of alumina scale observed (Figure 8(f)). The polished IN 693 specimen formed an outer uniform Cr_2O_3 oxide scale (Figure 8(g)). On the 1000 grit specimen, local areas of alumina scale could be found (Figure 8(h)), while on the 80 grit surface, most of the sample surface was covered by Al₂O₃ with numerous islands of external Cr_2O_3 (Figure 8(i)).

2. Cross-sections

At 1000 °C, all three studied alloys tended to produce mainly the type II oxidation morphology according to Giggins and Pettit,^[11] i.e., an external Cr_2O_3 layer followed by internal precipitation of Al_2O_3 (Figure 9). IN 617 (Figure 9(a) through (c)) and 602 CA



Fig. 7—SEM/BSE elemental map obtained on the cross-sections of IN 617 (a, b), 602 CA (c, d), and IN 693 (e, f) after 48 h of air oxidation at 900 °C with: polished (a, c, e) and 80 grit ground (b, d, f) surface.

(Figure 9(d) through (f)) followed this pattern irrespective of surface preparation. However, for IN 693, the type II morphology was observed only for the polished specimen (Figure 9(g)). The samples ground using 1000 grit and especially 80 grit sandpaper developed a continuous sublayer of Al_2O_3 (Figure 9(h) and (i)). Furthermore, the 80 grit IN 693 specimen formed a much thinner oxide scale. This observation agrees well with the mass change data (Figure 2).

IV. DISCUSSION

The obtained results suggest that two typical types of oxide scale morphology for NiCrAl-base alloys as postulated by Giggins and Pettit^[11] were observed for the alloys under investigation, i.e., an external chromia scale followed by an internal oxidation zone (IOZ) of Al (type II) and external alumina scales sporadically overlaid by patches of Cr_2O_3 (type III). IN 617 exposed at both 900 °C and 1000 °C tends to produce exclusively the type II oxide scale morphology. At the same time, this alloy demonstrated an increase of the oxygen uptake with increasing surface roughness. The thickness of the external chromia scale was similar over the entire



Fig. 8—SEM/BSE images of the surfaces of IN 617 (*a* through *c*), 602 CA (*d* through *f*), and IN 693 (*g* through *i*) after 48 h of air oxidation at 1000 °C: polished surface (a, d, g), 1000 grit (b, e, h), and 80 grit (c, f, i).

temperature range. Hence, cold work introduced by grinding promotes internal precipitation of Al_2O_3 , most probably promoting diffusion of Al toward the surface over the introduced dislocations and GBs in the recrystallization subsurface zone.^[12]

Additionally, the outer chromia formed on IN 617 was thicker compared with 602 CA and IN 693. This effect can be related to the higher Ti content in the alloy and its participation in the oxidation process as shown in the GD-OES depth profiles (Figure 5(a) and (b)). Titanium is known to accelerate chromia growth, as demonstrated by Jalowicka *et al.*^[15]

At the scale–alloy interface of the outer chromia scale and base alloy, it was observed that Cr_2O_3 scale was impregnated with particles of metal originating from the IOZ. The metal was extruded outward owing to the volume change in the IOZ induced by the formation of Al_2O_3 in the internal oxidation zone. The metal extrusion was most pronounced for alloy 602 CA because of its high, borderline Al content. The volume fraction of Al_2O_3 in the IOZ grown on alloy 602 CA was approximately 0.27,^[10] approaching the commonly accepted value of 0.3 introduced by Rapp^[31] required for the transition from external to internal oxidation. For the alumna-forming alloys, namely 602 CA and IN 693, it was found that increasing the surface roughness facilitated the transition from internal to external alumina scale. Such an effect was previously reported by Nowak *et al.* and explained by the introduction of defects in the near-surface regions of the alloys.^[16]

The results obtained for IN 617 showed that the surface finish had virtually no effect on the oxide scale morphology in terms of the external scale thickness and IOZ depth. However, protruding ridges of chromia could be observed above the grain boundaries of the alloy. Such oxide ridges were reported by Bataillou et al.^[32] and interpreted as faster oxide growth above the grain boundaries due to faster transport of Cr over the alloy grain boundaries. This explanation has a weak point as the rate of diffusion-controlled processes such as oxide scaling during oxidation is limited by the slowest step, i.e., diffusion in Cr2O3, and cannot be accelerated by faster Cr supply over the GBs. A more plausible explanation for the ridges is oxide scale delamination over GBs followed by the formation of new oxide area, healing the void underneath the delaminated oxide (see voids in Figure 6(c)).^[33,34] Thermally grown Cr_2O_3 is known to be under compressive growth stress^[35–37] and may delaminate above GBs. As the ridges become less pronounced with increasing



Fig. 9—SEM/BSE images of the cross-sections of IN 617 (*a* through *c*), 602 CA (*d* through *f*), and IN 693 (*g* through *i*) after 48 h of air oxidation at 1000 °C: polished surface (a, d, g), 1000 grit ground surface (b, e, h), and 80 grit ground surface (c, f, i).

surface roughness, it is likely that the Cr_2O_3 scale adheres better to a rougher alloy surface, thereby preventing delamination. Further research is needed to verify this mechanism.

As can be seen in Table I, IN 617 contains 1.2 wt pct of Al and thus can oxidize exclusively internally, forming an IOZ. The IOZ depth is not affected by cold work. The IOZ depth is controlled by oxygen diffusion, i.e., oxygen permeability N_0D_0 .^[38] Apparently, cold work and recrystallization do not affect transport of the interstitial species such as oxygen.

At 900 °C, the IOZ width in alloy 602 CA diminished with increasing surface roughness, which is related to the partial transition from internal to external alumina scaling.

For alloy 693 exposed at 900 °C, the outer scale thickness decreased with increasing surface roughness as the chromia scaling was overtaken by α -alumina growth.

As mentioned above, the reason for such a transition from Cr_2O_3 to Al_2O_3 scaling is due to the introduction of large number of defects and stresses into the near-surface region by grinding, which can be relieved, e.g., by mechanical polishing. Higher concentration of defects and stresses in the near-surface region provokes recrystallization at high temperatures. To prove this

concept, samples with polished and 80 grit ground surfaces were oxidized for 1 h at 900 °C. The metallographic cross-sections were electrochemically etched to reveal the grain structure. The images of the etched cross-sections are shown in Figure 10(a) through (f). As can be clearly seen for all three alloys with ground surfaces, a thin fine-grained layer of structure immediately underneath the oxide scales is present (marked by red arrows in Figure 10(b), (d), and (f)). The depth of the recrystallized zones is marked by dashed yellow lines. In the polished samples such a zone is absent. Similar observations were made for samples after exposure at 1000 °C. To further verify the surface cold-working effect on the recrystallization process, samples of IN 617 with polished and 80 grit ground surfaces were heat treated for 1 h in vacuum (10^{-6} mbar) at temperatures of 900 °C and 1000 °C. Similar to the oxidized specimens described above, recrystallization zones were observed in the ground specimens heat-treated at both temperatures, while no recrystallization zone was found in the polished ones (Figures 11(a) through (d)). The recrystallization depth in the ground IN 617 specimen was larger at 900 °C (Figure 11(b)) compared with 1000 °C (Figure 11(d)). As diffusion is faster at 1000 °C, stress relaxation occurs



Fig. 10—SEM/BSE images obtained on the electrochemically etched cross-sections of IN 617 (a, b), 602 CA (c, d), and IN 693 (e, f) after 48 h of air oxidation at 900 °C with: polished (a, c, e) and 80 grit ground (b, d, f) surface (Color figure online).



Fig. 11—SEM/BSE images obtained on IN 617 with polished (a, c) and 80 grit ground (b, d) after heat treatment in vacuum at 900 °C (a, b) and 1000 °C (c, d).

faster, as was demonstrated at 1100 to 1200 °C for alloy 602 CA.^[39] The short-term exposures fully confirmed the recrystallization hypothesis (Figures 10 and 11).

At 1000 °C, IN 617 behaves in a similar manner as during oxidation at 900 °C; namely, it forms an external chromia scale and an internal oxidation zone of Al (Figure 12). However, a stronger mass change increase is observed with increasing surface roughness (Figure 2). Remarkably, grain boundaries of the recrystallized grains are decorated internally with Al₂O₃ precipitates under image analysis of the IOZ (Figure 13) performed to better quantify and understand this effect. The volume fractions of alumina were calculated using the algorithm in NIS-Elements 2.3 software. The results obtained are shown in Figure 14. It is clear that increasing the surface roughness results in a higher volume fraction of internally oxidized Al₂O₃. The observed trend is stronger at 1000 °C. As cold work promotes recrystallization, more internal Al₂O₃ is precipitated in the IOZ, which is reflected in the higher oxygen uptake for the ground samples compared with the polished ones.

In the case of alloy 602 CA, the type II oxide scale morphology was produced irrespective of the surface roughness.

Finally, alloy 693 containing 3.3 wt% formed a duplex oxide scale during oxidation at 1000 °C. A network of internal alumina healed by an internal sublayer was overlaid by chromia. The R_s of the outer Cr_2O_3 decreased with increasing surface roughness as well as the mass change. It is obvious that cold work promoted alumina scaling more rapidly and suppressed the transient oxidation of Cr.

At both temperatures, one can clearly see the effect of the Al content on the IOZ width. According to the classical theory of internal oxidation,^[40] the parabolic rate constant for IOZ growth, k_{PIOZ} , is inversely proportional to the Al content, N_{Al} . This effect is unequivocally visible only in the polished specimens (Figures 6 and 9) as the polishing releases mechanical stresses in the subsurface layer and eliminates defects and dislocations introduced by grinding.



Fig. 12—SEM/BSE images obtained on IN 617 with polished (a, c) and 80 grit ground (b, d) after air oxidation at 900 °C (a, b) and 1000 °C (c, d) for 48 h.



Fig. 13—SEM/BSE images of the cross-sections of: (a) polished, (b) 1000 grit, and (c) 80 grit ground IN 617 after air exposure at 1000 °C for 48h and their binarized images ((d), (e), (f) respectively) used for image analysis.



Fig. 14—Effect of cold work on volume fraction of Al_2O_3 in internal oxidation zone in IN 617 after 48 h of air oxidation at 900 °C and 1000 °C.

V. CONCLUSIONS

On the basis of the obtained results, the following conclusions can be made:

- Mechanical cold work introduced by grinding promotes selective oxidation of Al.
- Formation of external alumina was more pronounced at 900 °C compared with 1000 °C.
- Cold work suppresses chromia ridges over GBs, increasing the surface roughness and improving the oxide adhesion.
- Cold work increases the volume fraction of alumina in the IOZ, not affecting the IOZ thickness owing to the higher grain boundary density below the external chromia scale with more opportunities to nucleate Al₂O₃ in the IOZ.
- Cold-work-induced subsurface recrystallization is demonstrated to promote protective oxidation behavior and is beneficial for the oxidation resistance of NiCrAl-base alloys.

CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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