Significantly enhanced tensile strength and ductility in nickel aluminium bronze by equal channel angular pressing and subsequent heat treatment

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Abstract

Nickel aluminium bronze (NAB) was subjected to equal channel angular pressing at 400°C for up to four passes in routes B_A and C, respectively, followed by isothermal heat treatment with a view to improving the κ phase structures and tensile properties. The lamellar κ_{III} structure was completely broken after 4 passes in route B_A although route C was less efficient. Spheroidisation and coarsening of the highly deformed κ_{III} continued during heat treatment especially at $\geq 600^\circ\text{C}$. At 800°C, both the lamellar structure and the fine κ_{IV} particles transformed completely into a coarse globular morphology with no distinction between the primary and eutectoid α . Significant increases in strength were achieved by ECAP, reaching a maximum yield strength of 960 MPa with a good ductility of ~14%. Heat treatment after ECAP was shown to considerably improve tensile ductility to > 30% while keeping the strength high at ~700 MPa, a significant enhancement compared to the as-received NAB.

Keywords: Equal channel angular pressing, severe plastic deformation, lamellar structure, spheroidisation, tensile properties.

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1. Introduction

Nickel aluminium bronze (NAB) is a copper based alloy which finds wide use in the marine industry thanks to its unique combination of high strength, corrosion resistance and protection against biofouling [1, 2]. The impressive corrosion resistance of aluminium bronzes has been linked to the quick formation of a protective sub-layer of Al_2O_3 which hampers ionic transport of cuprous ions to the surface layer of Cu_2O [1]. Compared to the single α -phase alloys, additions of up to 5 wt.% Ni and 5 wt.% Fe in NAB improve both strength and corrosion resistance by allowing higher concentrations of aluminium whilst suppressing formation of the adverse γ_2 phase which accelerates corrosion [2]. However, these additions also lead to a complex microstructure consisting of several distinct phases including the large rosettes of Fe_3Al denoted as κ_{I} or κ_{II} (distinguishable by size and location [3]), the lamellar NiAl based κ_{III} phase forming a eutectoid structure, and the fine Fe_3Al based κ_{IV} precipitates in the primary α matrix [2, 3]. These intermetallic phases cause several complications in both mechanical and corrosion performances. Easy paths for crack propagation may be found along κ_{I} and κ_{II} boundaries [4], limiting the ductility of cast components. The larger κ_{I} and κ_{II} rosettes are also known to pull out during cavitation attack under turbulent flow conditions [5]. More significantly, selective phase corrosion along the α - κ_{III} interface leads to deterioration of the surface [1, 6], and can penetrate deep beneath the surface under crevice corrosion conditions [7]. It is expected that by eliminating the lamellar structure of κ_{III} and strengthening the α matrix, both corrosion and cavitation properties can be improved. Wharton *et al.* made such observations in wrought NAB which contained little κ_{III} [1], although no direct comparison between the corrosion properties of wrought and cast NAB was made.

With the advent of severe plastic deformation (SPD) techniques, new possibilities for enhancing the corrosion resistance of NAB become available [6, 8, 9], with the added benefit of ultra high strength through grain refinement [10-14]. This idea is supported by a recent study on the surface modification of NAB by friction stir processing [6], in which the corrosion rate was

significantly decreased through a combination of grain refinement and homogenisation of the κ -phases. However, the refinement offered by friction stir processing is limited to the surface layer and no strengthening is provided to the bulk of the material. Equal channel angular pressing (ECAP), on the other hand, is now widely accepted as a SPD process for the production of bulk ultrafine grained materials [10]. ECAP has also been shown to be effective in refining lamellar structures, either through shear based transformations [15] or spheroidisation during post-ECAP heat treatment [16-18]. To date, no investigations on the mechanical properties of ultrafine grained NAB have been published although the properties of other aluminium bronzes processed by ECAP are promising. In a series of studies by An *et al.* [12-14], the yield strength of an aluminium bronze increased significantly from ~70 MPa in the coarse grained material, to over 700 MPa after ECAP. Such strengthening in NAB would be highly beneficial, not only in boosting the performance of the NAB components, but also in potentially replacing some stainless steel components which may suffer from galvanic corrosion. However, limited dislocation storage in ultrafine grained materials resulted in little or no work hardening and a tendency for the early onset of plastic instability [10-12, 19]. The instability can be mitigated by post ECAP heat treatment at the cost of strength, but this does not always result in an increase in ductility [13, 14]. The purpose of this paper is threefold: (1) to eliminate the lamellar structure in NAB using ECAP, (2) to further modify the microstructure through the use of post-ECAP heat treatment, and (3) to achieve a combination of high strength and good ductility in NAB through the use of ECAP and heat treatment.

2. Experimental material and procedures

A cast NAB plate with a composition of Cu–8.8 Al–4.4 Fe–5.2 Ni–1.1 Mn was supplied by VEEM Australia, after heat treatment at 675°C for 6 h. Such heat treatment is necessary to fully transform any retained β' phase into $\alpha + \kappa_{III}$, and is known to improve corrosion resistance [2]. The plate was machined into bars of 8 × 8 × 10 mm for ECAP. These specimens were wrapped in

graphite and inserted into a 90° ECAP die with a cross section of 9 × 9 mm and inner and outer curvatures of 5 and 3 mm, respectively. The die was then heated to 400°C. After the temperature stabilised, pressing was conducted at 5 mm/min. In addition to a forward plunger in the entrance channel, a secondary back plunger was placed in the exit channel to provide a constant back pressure of 50 MPa during ECAP. Samples were pressed to a maximum of 4 passes using routes B_A (alternate 90° rotation of the sample between passes) and C (180° rotation between passes), respectively and quenched in water upon retrieval from the die. The ECAP processed specimens were sectioned into small samples for a series of isothermal heat treatments at temperatures between 300 and 800°C for a duration of 1 h, followed by water quenching.

Vickers microhardness (HV) was measured using an indenter load of 500 g and a dwell time of 15 s. Microstructural analysis was carried out using scanning electron microscopy (SEM) on samples prepared using standard metallography procedures. Tensile specimens possessing a gauge length of 6 mm and a cross section of 2 × 3 mm were tested at room temperature with an initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ and the displacement measured using a laser extensometer.

3. Results

3.1 Microstructures

The as-received NAB microstructure is presented in Fig. 1 showing the eutectoid structure containing κ_{III} lamellae, large κ_{II} rosettes mostly located inside the eutectoid region, and fine precipitates of κ_{IV} in a matrix of primary α . The lamellae in the as-received material were randomly oriented as κ_{III} is known to grow perpendicular to the primary α boundaries [2]. Fig. 2 depicts the microstructure after one pass of ECAP, with the lamellar orientations becoming highly directional. In particular, much of the lamellar κ_{III} was fragmented (arrow 1 and inset in Fig. 2a) and formed streams oriented close to 22.5° to the longitudinal direction of the exit channel (Fig. 2a), coinciding

with the principal strain direction for an ideal, sharp-cornered 90° ECAP die [20]. Spheroidised κ_{III} , identifiable by the surrounding area of κ_{IV} free eutectoid α , was also observed between the streams of fragments (arrow 2 in Fig. 2a). Perpendicular to the principal strain direction, the κ_{III} lamellae remained straight in the larger colonies consisting of a high number of laths (hollow arrow in Fig. 2b.) but those in smaller colonies buckled with a similar distortion pattern between adjacent laths (solid arrows in Fig. 2b).

The microstructures after 4 passes with route C and route B_A are shown in Fig. 3 and Fig 4, respectively. After the 4th pass in route C (Fig. 3), the directionality seen after the first pass reverted to the random orientations in the as-received state. However, the lamellar structure was altered in different ways with some κ_{III} spheroidised (arrow 1), some κ_{III} , although fragmented, forming lamellae again after redundant straining (arrow 2 and inset), and others fragmented and dispersed, i.e. not re-forming lamellae (arrow 3). In addition, some κ_{II} particles appeared to have collided (arrow 4). In contrast, the lamellar structure was completely transformed into fragmented and spheroidised κ_{III} after four passes in route B_A (Fig. 4) with the fragmented κ_{III} streams maintaining the alignment with the principal strain direction. The homogeneity of the microstructure was significantly improved from the as-received material, with the κ_{III} particles now comparable in size to the fine κ_{IV} precipitates. The morphology of κ_{II} was also influenced, evolving from the round shape in the as-received material to highly irregular (arrows in Fig. 4).

The microstructures following one pass of ECAP and then heat treatment at 600, 700 and 800°C for one hour are shown in Figs. 5a, 5b and 5c, respectively (there was little change at temperatures below 600°C). Slight coarsening (arrow 1 in Fig. 5a) of the fragmented κ_{III} and perturbations (arrow 2 and inset) along the distorted lamellae occurred at 600°C, indicating the start of the spheroidisation process. At the higher temperature of 700°C, the undistorted lamellae (arrow 1 in Fig. 5b) remained with the majority of the κ_{III} fragments now perfectly rounded following spheroidisation and coarsening (arrow 2). Coarsening of κ_{IV} was also apparent, reducing the

number of fine precipitates in the primary α . At 800°C, little distinction between the primary and eutectoid α could be made as all of the fine κ_{IV} had coarsened into large particles leaving precipitate free α behind. The lamellar κ_{III} was also no longer recognisable, as it had coarsened into partially elongated particles (arrow 1 in Fig. 5c). In some cases, these particles were seen to have sharp, angular edges (arrows 2 and 3).

The microstructures following ECAP for 4 passes with route B_A and heat treatment at 600°C, 700°C and 800°C are shown in Figs. 6a, 6b and 6c, respectively. As the lamellar structure had already been broken during ECAP, only coarsening of the spheroidised lamellar fragments was observed at 600°C and 700°C. At 800°C, the microstructure was nearly identical to that after one pass and heat treatment (Fig. 5c), although the coarsened globular κ_{III} appeared slightly more angular.

3.2 Microhardness

The HV versus number of ECAP passes is plotted in Fig. 7. The hardness increased significantly from 210 HV in the as-received material to ~315 HV after the first pass. The microhardness of route C was seen to fluctuate with subsequent passes, becoming slightly softer on even numbered passes, and harder on odd passes. This was not apparent for route B_A , which maintained a high hardness until the fourth pass, where it was seen to drop. The variation in hardness after ECAP was relatively small, with a standard deviation for all samples of ~4.8. The hardness following heat treatment at various temperatures is shown in Figs. 8a and 8b for the two routes, respectively. Little change in hardness was observed below 400°C for all ECAP conditions. The S-shape curve indicative of the occurrence of recovery and recrystallisation processes is only well defined for the single pass sample, and the curves became more linear above 400°C with increasing passes. This is particularly the case for route B_A (Fig. 8b). At 800°C, all samples had softened below the hardness of the as-received material.

3.3 Tensile properties

The tensile stress-strain curves for NAB after ECAP for 1, 2 and 4 passes at 400°C are shown in Figs. 9a and 9b for routes C and B_A, respectively. A significant jump in strength was observed after the first pass, with the yield stress increasing from ~180 MPa in the as-received material to ~960 MPa, accompanied by a significant reduction in ductility from ~24% to ~5%. After two passes, the sample processed by route B_A showed a continued increase in strength and ductility, rising to ~970 MPa and ~6%, respectively. This was in contrast to route C, which showed a slight reduction in strength to ~950 MPa with only a minor change in ductility. The difference in ductility became greater after 4 passes, with route B_A leading to a considerably improved elongation of ~14% (mostly during strain softening). The large increase in ductility of the route B_A sample did not coincide with any significant drop in yield stress, which remained close to 960 MPa, although the UTS was noticeably lower. Again, little change was observed in route C, which maintained a yield strength and ductility of ~930 MPa and ~6%.

The tensile stress-strain curves after isothermal heat treatment at 450 to 700°C are shown in Figs. 10a, 10b and 10c for the materials ECAP processed for 1, 2 and 4 passes with B_A, respectively. A general trend, as expected, is that the strength was decreased and ductility increased as the heat treatment temperature was increased. However, heat treatment at the relatively low temperature of 450°C resulted in no improvement in ductility in the sample processed for 1 pass, and some moderate improvements in the samples processed for 2 and 4 passes. Although strength continued to drop with increasing temperature, the materials heat treated at 600 and 700°C exhibited superiority in both strength and ductility, especially in the 4-pass sample, compared to the as-received material. The strain softening behaviour observed in the sample processed for one pass was eliminated at all temperatures. However, temperatures of 500 and 550°C were required to eliminate work softening in samples after ECAP processing for 2 and 4 passes, respectively.

4. Discussion

4.1 Deformation behaviour of the lamellar κ_{III} phase

The streamed κ_{III} fragments (arrow 1 in Fig. 2a) and the spheroidised κ_{III} between them in the as-ECAP processed microstructure (arrow 2 in Fig. 2a) are unique. ECAP has been shown to cause deformation induced transformations in wholly lamellar microstructures such as eutectic Al-Cu [15] and high carbon steels [18], where such transformations become necessary to account for the geometric distortions to the multidirectional lamellar structure. However, in the duplex structures consisting of eutectic or eutectoid lamellar colonies and primary phase grains, such as the pearlite plus ferrite structures in low [16] to medium carbon steels [17], the occurrence of ECAP induced spheroidisation is significantly less. Instead, the cementite lamellae were either stretched into fragments or compressed into curled, wavy laths [16, 17], depending on the initial orientation of the lamellae to the shear plane. This fragmentation could only be caused by a lengthening along the lamellar plane, as evidenced by the observed necking along the stretched cementite fragments [16, 17]. Indeed, similar morphologies of κ_{III} can be seen following ECAP deformation in the present investigation, although necking of the κ_{III} is not readily observed (inset in Fig. 2a). In this case, the ductility of the eutectoid phase should be taken into account as NiAl (on which the κ_{III} phase is based) shows little ductility at high strain rates [21, 22]. This is in contrast to the ECAP of a Cu-Ag alloy, where the softer and more ductile Ag precipitates were continually stretched into fine fibres after 4 passes in route A [23]. Furthermore, as analysed in [20], the maximum stretching occurs in the principal strain direction, and should coincide with the alignment of the fragmented streams, as observed here. Hence, both lengthening and low ductility in the eutectoid lamellae are required for their break down via fragmentation. On the other hand, maximum shortening occurs perpendicular to the principal strain direction. In both lamellar cementite and lamellar κ_{III} , such a contraction results in buckling of the laths, causing it to become warped or wavy. However, the extent of buckling in κ_{III} is much greater than in cementite. This is possibly due to the difference in their lamellar spacings. In the as-received NAB, the lamellar spacing of κ_{III} was ~ 650 nm,

compared to 160 nm for the cementite in [16]. As seen in Fig. 2b (solid arrows), the distortion in one lath is also experienced by neighbouring laths. When the lamellar spacing is reduced, the distortion must be shared over a larger number of laths, and this has been shown to increase the deformation resistance of the colony (as seen in the lower section of Fig. 2b).

4.2 Spheroidisation of the lamellar κ_{III} phase

The consequence of the buckling is the creation of many highly curved sections of the κ_{III} lamellae. It is well established that curved surfaces intrinsically have a higher chemical potential than flat ones [24], and thus are less stable. When the lamellae are buckled during ECAP, some sections with very high curvatures may possess enough driving energy to undergo spheroidisation even at the low ECAP temperature of 400°C. This curvature-enhanced spheroidisation would continue during the subsequent heat treatment. Although at 600°C the overall changes appeared not significant, the highly curved segments found in the as-ECAP processed state are no longer existent. Increasing the temperature to 700°C, only the straight lamellae survived the spheroidisation process. (The microstructure at 800°C cannot be entirely attributed to spheroidisation and will be discussed in the next section.) It can be concluded, therefore, that the streams of spheroidised κ_{III} are actually a result of severe buckling which produces curvatures greater than needed by spheroidisation at 400°C, leading to the break-up of the lamellae. The additional thermal energy provided by post ECAP heat treatment then allows the less deformed lamellae to spheroidise. Moreover, it should be remembered that the production of a large number of dislocations and vacancies during ECAP may also increase the speed of diffusion, accelerating the spheroidisation process.

4.3 Dissolution of the κ -phases at 800°C

When heat treated at 800°C, vastly different microstructures resulted. These microstructures are similar to the one obtained in a cast NAB after heat treatment at 860°C for 3 h and almost identical to the one treated at 840°C for 72 h [2]. In all the cases, little distinction could be made

between primary and eutectoid α , with the κ -phases all existing in coarse globular morphologies. The coarsening of the fine κ_{IV} precipitates into large globules may be due to its transformation from κ_{IV} to a high temperature κ_V phase [2]. Above 675°C, the predominantly iron rich κ_{IV} begins to absorb nickel from its surroundings, eventually achieving equal amounts of the two elements. In doing so, the originally globular κ_{IV} transforms into a short lath morphology identified as κ_V . Whereas κ_{IV} and/or κ_V coarsens gradually as the temperature is raised from 600°C to 800°C, the κ_{III} phase shows a sudden change from fine fragments/spheroids to large, irregularly shaped particles at 800°C. On closer inspection, the irregular shape appears to be formed by the borders of the surrounding α grains. The answer to this inconsistency may lie in the dissolution temperature of κ_{III} , thought to be close to 825°C [2]. At this temperature, the necessary diffusion for particle drag by grain boundaries may be achieved, and this has been shown to occur for precipitates similar in size to the finely fragmented κ_{III} [25]. In heat treated high carbon steel processed by ECAP, it was shown that the spheroidised cementite was preferentially located at the triple points of the surrounding ferrite grains [18]. The same preferred location may also apply to the κ_{III} fragments and spheroids. In this case, if particle drag were to occur through grain boundary migration, the fragments of κ_{III} would eventually coalesce as these triple points merge. This process would continue until the particle becomes too large to move. Coarsening of the coalesced κ_{III} could still continue through Ostwald ripening, and the growth may lead nearby clusters to impinge on one another at the interface [26]. Since the migration of the κ_{III} phase occurs at the boundaries of the α grains, it is shaped by them, resulting in the microstructure shown in Figs. 5c and 6c.

4.4 Effect of ECAP and heat treatment on mechanical properties

The mechanical properties of ECAP processed NAB are dependent on both the κ phase and grain structures. In particular, an effective refinement of the lamellar phase is crucial to enhance ductility in NAB. This is most clearly evidenced by the differences in mechanical properties between samples processed by route C (Fig. 9a) and route B_A (Fig. 9b). Although both display

similar HV values (Fig. 7), the superior ability of route B_A to produce a homogeneous microstructure without any lamellar κ_{III} leads to a significantly greater ductility after 4 passes compared to route C (Fig. 9). In route C, the remaining lamellae would provide opportunities for stress concentrations and easy paths for fracture, both contributing to early failure. In contrast, when single phase aluminium bronze was ECAP processed [12], slight decreases in ductility were observed with increasing number of passes (route B_C though). This implies that the increase in ductility gained through the homogenisation of the NAB microstructure is greater than the ductility loss due to a higher level of defects as a result of SPD. This point is further emphasised when comparing the tensile properties in the as-received NAB to those after 4 passes of ECAP in route B_A and then heat treatment at 700°C. The ECAP processed and heat treated sample is not only much stronger, but also possesses a greater ductility, ~8% higher than the as-received material.

The decrease in yield strength or hardness after heat treatment was less significant with increasing number of passes, especially with route B_A (~430 MPa, ~440 MPa and ~570 MPa in the 1-, 2- and 4-pass samples after heat treatment at 700°C). This is most likely due to the different softening processes. Significant recrystallisation may have occurred in the 1-pass sample as evidenced by the S-shaped curve in Fig. 8b. In contrast, the more linear curve for the 4-pass sample suggests grain growth as the dominant process. Detailed grain structure investigation is in progress to verify this point.

ECAP processed NAB is stronger than the ECAP processed aluminium bronze [12], and comparable in strength to aluminium bronze processed by high pressure torsion (HPT) [14]. The aluminium bronze in both cases had been processed at room temperature, significantly lower than the 400°C processing temperature in the present work. This increased strength may have resulted from a combination of increased solid solution content in the α phase and strengthening originating from the κ phases. After heat treatment, ECAP processed NAB retains the same strength as that of the as-ECAP processed aluminium bronze but does not suffer from strain softening which is

expected in HPT processed aluminium bronze due to its much smaller grain sizes. Therefore, this current study points to the potential of using a combination of ECAP and heat treatment for producing high strength NAB with good ductility.

5. Summary and conclusions

- (1) Cast NAB was processed by ECAP at 400°C for up to 4 passes using routes B_A and C, respectively. Route B_A proved to be more efficient at breaking the lamellar structure and producing a fine dispersion of refined κ_{III} uniformly distributed in the α matrix, leading to a significantly enhanced yield strength of 960 MPa with a good tensile elongation of ~14% after 4 passes.
- (2) The ECAP processed NAB was subsequently subjected to isothermal heat treatments at temperatures between 300 and 800°C. Although it had caused increasing drop in strength with increasing temperature, the heat treatment considerably improved the ductility and eliminated the work softening when conducted at $\geq 500^\circ\text{C}$. The best combination of tensile properties was achieved in NAB processed for 4 passes with B_A after heat treatment at 600-700°C, with yield strength of 500 to 700 MPa and elongation of $> 30\%$, greatly exceeding < 200 MPa and ~25% in the as-received NAB.
- (3) The break-down of the lamellar κ_{III} was initiated during ECAP through fragmentation of the laths under tension and spheroidisation at highly curved sections. The spheroidisation process continued during heat treatment, especially at $\geq 600^\circ\text{C}$, leading to the complete disappearance of the lamellar κ_{III} in the material processed for 4 passes in B_A.
- (4) At the highest heat treating temperature of 800°C, both the fragmented and still lamellar κ_{III} in all the ECAP processed materials spheroidised and coarsened into a globular morphology

located between the α grains, probably assisted by grain boundary migration. The fine κ_{IV} precipitates were also coarsened and may have transformed into the high temperature κ_V phase.

- (5) No softening occurred during heat treatment at $\leq 400^\circ\text{C}$ in all the materials and the three-stage softening due to recovery, recrystallisation and grain growth was only obvious in the materials processed for one pass. With increasing number of passes, it seemed that grain growth became the more dominant softening process, especially in the material processed for 4 passes in B_A .

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Figure captions

- Figure 1 Microstructure of the as-received NAB, showing various κ phases.
- Figure 2 Microstructures of NAB after ECAP at 400°C for 1 pass (the longitudinal direction of the specimen is horizontal), showing (a) fragmented (arrow 1 and inset) and spheroidised κ_{III} (arrow 2) and (b) largely undeformed laths (hollow arrow) in a large κ_{III} lamellar colony and buckled ones (solid arrows) in a small κ_{III} lamellar colony.
- Figure 3 Microstructure of NAB after ECAP at 400°C for 4 passes in route C (the longitudinal direction of the specimen is horizontal), showing a partially transformed κ_{III} structure with some spheroidised (arrow 1), some fragmented but then re-forming lamellae (arrow 2 and inset) and others fragmented and dispersed (arrow 3). Some κ_{II} particles appeared to have collided (arrow 4).
- Figure 4 Microstructure of NAB after ECAP at 400°C for 4 passes in route B_A (the longitudinal direction of the specimen is horizontal), showing a completely transformed κ_{III} structure with both fragmented and spheroidised κ_{III} streams aligning in the direction of the principal strain, with the κ_{II} particles becoming irregularly shaped (arrows).
- Figure 5 Microstructures of NAB after ECAP at 400°C for 1 pass, followed by one-hour heat treatment at (a) 600, (b) 700 and (c) 800°C, showing increasingly transformed κ phases. (The arrowed features are explained in the text.)
- Figure 6 Microstructures of NAB after ECAP at 400°C for 4 passes in B_A, followed by one-hour heat treatment at (a) 600, (b) 700 and (c) 800°C, showing completely spheroidised and coarsened κ_{III} at all the temperatures.
- Figure 7 Vickers microhardness versus the number of passes following ECAP at 400°C with routes C and B_A, respectively.
- Figure 8 Vickers microhardness versus the isothermal heat treatment temperature for samples ECAP processed at 400°C for 1, 2 and 4 passes following (a) route C and (b) route B_A.
- Figure 9 Tensile stress-strain curves for NAB after ECAP at 400°C for 1, 2 and 4 passes following (a) route C and (b) route B_A. The curves for as-received NAB are also presented for comparison.
- Figure 10 Tensile stress-strain curves for NAB after ECAP at 400°C for (a) 1, (b) 2 and (c) 4 passes with route B_A, followed by heat treatment at various temperatures for one hour. The curves for as-received NAB are also presented for comparison.

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