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Microstructure, mechanical and corrosion properties of cryorolled-AA5052 at various solution treatment temperatures

N M Anas, T E Abioye1,2, A S Anasyida1 ©, B K Dhindaw1, H Zuhailawati1 and Azzura Ismail1

1 Structural Niche Area, School of Materials & Mineral Resources Engineering, Universiti Sains Malaysia, Engineering Campus 14300 Nibong Tebal, Pulau Pinang, Malaysia
2 Industrial and Production Engineering, Federal University of Technology Akure, PMB 704, Akure, Ondo State, Nigeria
3 School of Minerals Metallurgical and Materials Engineering, IIT Bhubaneswar, Bhubaneswar 751007, India
4 Department of Materials Engineering and Design Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia 86400 Batu Pahat, Johor, Malaysia

E-mail: anasyida@usm.my

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**Abstract**

Initial solution treatment (ST) of aluminium alloys prior cryorolling (CR) enhances the ultrafine grain structure formation hence, improving their mechanical and corrosion properties. However, determining the appropriate ST temperature is crucial to obtain the optimum properties. In this work, ST followed by cryorolling of AA 5052 alloy was performed at varying temperatures; 480 °C, 510 °C and 540 °C. Mechanical and microstructural characterization of the cryorolled samples with and without ST and the base metal were performed using Vickers hardness tester, universal tensile testing machine, FESEM, EBSD and HRTEM. All the ST cryorolled samples showed the finer structure and better mechanical and corrosion performances than those without ST and the base metal. The smallest crystallite size and lattice strain achieved were 24.88 nm and 3.86 × 10⁻³ respectively at 540 °C. The hardness, yield and tensile strength increased with increasing the ST temperature with the highest values of 85.2 Hv, 254.82 MPa and 303.35 MPa, respectively, at 540 °C. Cryorolled sample solution treated at 540 °C also showed the highest corrosion resistance based on low corrosion current and high corrosion potential. The work gives the appropriate ST temperature at which improved microstructure, mechanical and corrosion properties of the cryorolled AA5052 can be obtained.

**1. Introduction**

Cryorolling (CR) is a severe plastic deformation process that consists of dipping a metal in liquid nitrogen (−196 °C) for a certain period of time prior to each rolling pass [1]. Compared with the well-known severe plastic deformation processes (SPD) such as accumulative roll bonding and high-pressure torsion, CR is the only process which is suitable for producing thin sheets in large scale production on a continuous basis and gives superior grain refinement and bi-modal grain structure. This often results in enhanced strength and ductility [2]. Also, CR is increasingly being utilized for the production of ultrafine-grained (UFG) structures with networks of high dislocation density in metal alloys hence, making the alloys suitable for structural applications in the form of sheets and plates. Rolling of materials at cryogenic temperature suppresses dynamic recovery and the density of accumulated dislocations reaches a higher steady-state level as compared to conventional rolling [3]. According to Li et al [4] deep-cryogenic treatment drastically changed the mechanical properties of aluminium alloys by refining the grain size and altering the distribution of fine precipitates. More so, CR of aluminium alloys broadens the materials’ applications in the manufacturing industries because of the possibility to simultaneously produce aluminium alloy sheet with UFG microstructures which is otherwise unachievable or costly to achieve with other SPD processes [5]. As a result, obtaining UFG structure in aluminium alloys through CR process has received significant attention because of the possibility of achieving superior geometrical, mechanical and corrosion properties in a single process.

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Often, metals require optimum pre-heat treatment before they undergo cryogenic deformation. The absence of any pre-heat treatment could lead to inadequate mechanical properties. Most of the researchers have performed pre-treatment before the material underwent cryogenic deformation [6]. Enhanced pre-heat treatment processes followed by CR could provide several numbers of strengthening mechanism for most of the metals, especially aluminium and its alloys. Hence, it is crucial for pre-heat treatment to be performed in order to provide the initial favourable condition that allows easy cryogenic deformation of metals. A lot of studies have focused on the effect of a package treatment which consists of solution treatment, cryogenic deformation, followed by post heat treatment such as ageing or annealing process. These have been well reported for various aluminium alloys such as AA 2014 [7], AA 6063 [8, 9], AA 7075 [10], etc The study on the development of UFG AA 5xxx by cryorolling process with the enhanced pre-heat treatment condition has not received adequate attention. Particularly, the effects of solution treatment on the mechanical properties and deformation mechanism of AA 5xxx series has not been reported.

Principally, the temperature of solution treatment could affect the number of solute elements that dissolves in supersaturated solid solution. A high temperature of solution treatment is essential for complete dissolution of the solute element to introduce maximum solid solution strengthening. The improvement of mechanical properties is also due to the solid solution strengthening via dissolution of more solute element [11, 12]. In aluminium alloys, the solution treatment temperature is below the solvus line and usually limited to below 600°C because the solution treatment at higher temperatures could lead to partial melting of aluminium matrix which results in diminishing of mechanical properties. Prasad and Dan [13] also claimed that the low temperature of solution treatment could result in the incomplete dissolution of undissolved soluble phases and a decreased level of homogenization of the matrix. However, a very high temperature of solution treatment leads to partial melting. Hence, the solution treatment temperature is one of the important factors since the strengthening effect of aluminium alloys is determined by the degree of solute supersaturation in solid solution. Therefore, solution treatment’s temperature is a crucial parameter needing a systematic study.

In this study, CR of AA 5052 that was solution treated at varying temperatures (480 °C–540 °C) and that without solution treatment was performed. The microstructure, mechanical and corrosion properties of the entire cryorolled samples including the base metal were investigated. The results were analysed so as to determine the optimum solution treatment (ST) temperature and the effects of the ST temperature on the mechanical and corrosion properties of the cryorolled alloy.

2. Experimental procedure

A commercial AA 5052 in sheet form used for structural applications was selected for the present study. The chemical composition of this aluminium alloy is 1.83 wt% Mg, 0.37 wt% Si, 0.33 wt% Fe, 0.14 wt% Cr, and remaining is Al. Samples with 1.2 × 20 × 50 mm dimensions were cut from sheets and solution treated at 480 °C, 510 °C and 540 °C with a heating rate of 10 °C min⁻¹. They were soaked for 6 h at solution treatment temperature followed by quenching in the water (25 °C). After solution treatment, the samples were cryorolled to 30% reduction in thickness. The samples were then characterized to examine the effect of solution treatment temperature. The crystallite size and lattice strain of cryorolled AA5052 were analyzed using X-ray Diffraction (XRD) at the scanning angle of 2θ from 10° to 90°. From the XRD patterns obtained, the crystallite size and lattice strain ((ε²)½) can be calculated using the Scherrer equation. The Scherrer equation is shown in equations (1) and (2) respectively [14].

\[
\text{Crystallite size (nm), } \tau = \frac{k\lambda}{\beta \cos \theta} \tag{1}
\]

\[
\text{Lattice strain, } \eta = \frac{\beta}{4 \tan \theta} \tag{2}
\]

\[
\rho = \frac{3\sqrt{2\pi}}{\tau b} (\eta^2)^{1/2} \tag{3}
\]

where k is a dimensionless shape factor and has a typical value of about 0.9, λ is the x-ray wavelength, typically 1.54 Å, β is the line broadening at half of the maximum intensity (FWHM) in radians, θ is the Bragg angle and b is the burgers vector. These data were obtained from the analysis using X’ Pert HighScore Plus 2.1.

Thereafter, Vickers hardness was performed on the cryorolled samples using 100 gf and a dwell time of 10 s. All the samples were ground before hardness measurement. Ten readings were taken for each sample. A tensile test was conducted using the Instron universal testing machine. The tensile test was performed on the specimen along the cryorolling direction according to the ASTM E8 with 50 mm gauge length. The test was conducted with a strain rate of 1 mm min⁻¹ until the specimen failed. Three test pieces for each of the sample were tested and average values were taken. The result was recorded through a stress-strain graph. The ultimate tensile strength, yield strength, elongation and tensile strain were obtained from the tensile test. After the tensile test was conducted, each fractured sample with its representative fracture region was characterized using scanning
electron microscope (SEM) to reveal the fracture modes. High-resolution transmission electron microscope (HRTEM), model Technai F200 was used to obtain a high-quality image of the morphology of the grain structure of some selected samples. In order to study the possible mechanism of strengthening, electron backscattered diffraction (EBSD) characterization was done for the samples having the best combination of properties using Bruker QUANTAX CrystAlign EBSD.

Corrosion behaviour of the cryorolled samples was characterized by measuring corrosion potential, corrosion current density and corrosion rates from the polarization curves by Tafel extrapolation using NOVA software. Firstly, the sample was partially immersed in the electrolyte with a depth of 20 mm. The contact areas of all the samples are 200 mm². The potentiodynamic polarization measurements were conducted by applying an external potential to the working electrode (WE) and scanned over a predetermined potential range from −2.0 V to 2.0 V while the current response generated was measured. The slow scan rate of 0.0050 V s⁻¹ was used in these experiments so as to achieve steady-state conditions.

3. Results and discussion

3.1. Variation in hardness and strength of CR AA 5052

Figure 1 shows the effect of various solution treatment (ST) temperatures on the hardness of the cryorolled AA 5052. The hardness of the solution treated cryorolled (ST-CR) alloy increased from 75.9 HV₀.₁ to 85.2 HV₀.₁, showing an increment of about 12.3%, as the ST temperature increased from 480 °C to 540 °C. The hardness of the cryorolled alloy solution treated at 540 °C shows an improvement of about 13.6% and 58.6% when compared to that of the cryorolled alloy without solution treatment and as-received alloy respectively.

The enhancement of hardness properties of ST-CR sample at various pre-treatment temperatures can be due to the solid solution strengthening which depends on the degree of dissolution as well as the concentration of solute element in the solid solution [10]. The dissolution of the solute element was expected to be higher as the temperature of the solution treatment increases [15]. Thus, at high solution-treated temperatures, maximum dissolution of the solute element or particle dissolution will take place [12]. The solute atom present in the matrix of the aluminium alloy will act as an obstacle and effectively suppress the cross slip or climb of dislocations during dynamic recovery. In addition, the improvement in hardness properties could be due to the large dislocation density produced in the samples during the cryorolling process [16].

Figure 2 shows stress-strain curve of cryorolled Al 5052 at different pre-solution treatment temperatures. The ultimate tensile strength (UTS) of the cryorolled sample improved after solution treatment. From stress-strain curve in figure 2, formation of Portevin–Le Chatelier effect (PLC) can be clearly seen for all the sample. PLC effect is caused by the instabilities of dislocation motion during continuous deformation, hence plastic deformation also unstable which was depicted by the serrations of the curve. It usually associated with local plastic shear in slip bands and sometimes propagating deformation bands which caused corrugations of the originally smooth specimen surface and may give rise to early failure of the components [17]. Dierke et al [18] also stated that under a constant strain rate and temperature, the instability appears as serrations in the stress–strain curve with a characteristic range of frequency and amplitudes of stress drops.

Figure 3 presents the tensile properties of the as-received AA 5052, cryorolled AA 5052 without solution treatment and those were solution treated at varying temperatures. It is obvious that the tensile strengths (ranging between 292–303 MPa) and yield strengths (ranging between 245–255 MPa) of the entire solution
treated cryorolled AA 5052 samples are higher than that of the cryorolled alloy without solution treated and much higher than the as-received alloy. The tensile strengths of the cryorolled alloy without solution treated and as-received alloy are 289 MPa and 260 MPa respectively while their yield strengths are 238 MPa and 186 MPa respectively. This shows that cryorolling process enhanced while solution treated further enhanced the tensile strength and yield strength of the as-received alloy. A similar phenomenon was reported by Nageswara et al\(^\text{[19]}\) during the cryorolling followed by warm rolling of Al–Mg–Si alloy. The tensile strain of the entire solution treated cryorolled AA5052 alloys are at least 67% lower than that of the as-received alloy but slightly higher than that of the cryorolled alloy without ST. The interpretation of this is that cryorolling significantly reduced the ductility of the alloy but the ductility was slightly improved with the solution treatment.

Comparing the solution treated cryorolled AA 5052 samples, the tensile properties increased as the solution treatment temperature increased. The tensile strength, yield strength and tensile strain of the solution treated cryorolled alloy increased by about 4%, 4% and 9% respectively as the solution treated temperature increased from 480 °C to 540 °C. The significant improvement in strength might be attributed to solid solution strengthening. The solute elements are dissolved inside the aluminium matrix in the substitutional sites during solutionizing and quenching of the AA 5052 alloy. The strength of the alloy was enhanced due to variations in size or valency of the solute and parent atoms\(^\text{[10]}\). Al-Marahleh\(^\text{[20]}\) has also claimed that the dissolved solute atoms after cooling formed strain fields which acted as obstacles to impede dislocation movement. The strain field created in the solution treated sample was suppressed due to the dynamic recovery during cryorolling process. Therefore, the strength of the solution treated sample is expected to increase after cryorolling process. As mentioned earlier, the dissolution of the solute element has been established to increase with increasing temperature of the solution\(^\text{[12]}\). Therefore, maximum dissolution of solute element or particle was expected at higher temperature hence an increase in the strength of the solution treated-CR alloy as the solution treatment temperature increased.
However, the lower ductility shown by the entire cryorolled samples, compared with the as-received AA 5052, can be attributed to insufficient strain hardening response which limits the capability of the material to propagate additional dislocations [21]. The slight increment exhibited by the ST-CR samples might be due to the increase in work-hardening rate. In order to further support the strengthening mechanisms outlined above, HRTEM, EBSD and X-ray diffraction studies were carried out.

3.2. HRTEM and EBSD micrograph of CR AA 5052

Figure 4 presents the HRTEM micrographs of the cryorolled solution treated sample at 540 °C that achieved the highest hardness, yield and tensile strength. The heavily deformed solution treated sample has a high density of dislocations generated during deformation. Solution treated sample after cryorolling as shown in figures 4(b) and (c) showed heavily deformed microstructure with high dislocation density. The existence of submicron grain structure can be seen in figure 4(a) at the magnification of 6.3k with grain size in the range of 0.4–0.75 μm. The nano-sized grain structure can also be observed in figure 4(b) at the magnification of 17.5 k, with a length of the grains in the range of 600–900 nm and the width about 85–500 nm. Figure 4(c) at the magnification of 255k revealed in some cases elongated nano-sized grain structure with a width of about 40 nm. Thus, cryorolled sample solution treated at 540 °C contains the combination of the submicron-sized and nano-sized grain structure. This combination of size and lengths obtained perhaps satisfies the criteria of UFG structures.

The cryorolled sample solution treated at 540 °C was also characterized by EBSD analysis. The green and blue colours seen in the EBSD maps in figure 5(a) indicated the location of high angle grain boundaries (≥15°) and low angle grain boundaries (1.5°–15°) respectively. The highest fraction of low angle misorientation boundaries of 3° observed in figure 5(b) is about 8.4 × 10³ number of grains. The distribution of high angle misorientation boundaries was observed only in about 0.3 × 10³ number of grains. The high fraction of low angle grain boundaries and a large amount of dislocation density observed in the solution treated cryorolled samples was due to the effective suppression of dynamic recovery [22]. The combination of both low and high angle grain misorientation boundaries as seen from EBSD results and accumulation of dislocation density in the cryorolled samples were vital in enhancing mechanical properties.
3.3. X-ray diffraction analysis of CR AA 5052 at various solution treatments

The XRD patterns of cryorolled samples at different solution treatment temperatures are presented in figure 6. The XRD patterns showed that the peak of (2 0 0) plane was dominant among others. This finding is consistent with the work done by Krishna et al. [23] where the solutionized sample exhibited a dominant peak of (2 0 0) plane. It was observed that there was no change in peak position. However, peak broadening and reduction in intensity were observed as the temperature of the solution treatment was increased.

As a result, the crystallite size and lattice strain at different ST temperatures were calculated using Scherrer equations (see equations (1) and (2)). The procedure involved in this calculation is detailed in the previous work of the authors [2]. The results, as presented in figure 7, showed that the crystallite size is inversely proportional to the lattice strain. In this study, the smallest crystallite size (24.88 nm) and the highest value of lattice strain \(3.86 \times 10^{-3}\) were achieved for the cryorolled sample solution treated at 540 °C. It can also be noted that as the solution treatment temperature increased, the crystallite size reduced and the lattice strain increased. This shows that the reduction of crystallite size is solution treatment temperature dependent. The cryorolled sample solution treated at 540 °C has the highest calculated lattice strain which implied that the deformation of this sample resulted in better suppression of dislocation movement.

The dislocation density was calculated based on XRD results as shown in table 1. The dislocation density increases with solution treatment temperatures. The dislocation density has the highest value of \(40.8 \times 10^{14} \text{ m}^{-2}\) at 540 °C. The suppression of dynamic recovery during rolling at cryogenic temperature increase the dislocation density.
3.4. Fracture surface of CR AA 5052 at various solution treatments

The fracture surface of the solution treated cryorolled samples after tensile testing were examined under SEM as shown in figure 8. The micrograph displayed that all the samples were fractured in a ductile manner, comprising of well-developed dimples over the entire surface as well as a rough and deep surface inside the dimples. The average dimple size of solution treated sample at 480 °C, 510 °C, and 540 °C are 3 ± 0.4 μm, 2 ± 0.5 μm and 1 ± 0.6 μm respectively. The size of dimple decreased with the rise in solution treatment temperature. Also, the amount of small dimples (microvoids) increases with the increasing of solution treatment temperature. The cryorolled sample solution treated at 540 °C seems to have more amount of dimples and some features of microvoid coalescence (dimpled rupture). The number of small-sized dimples increases and the depth of dimples becomes relatively shallow. Thus, this sample has a higher ductility compared to other samples. Jamaludin et al [24] claimed that the high ductility can be explained by observing the small and deep dimples on the fracture surface of the sample.

3.5. Corrosion properties of CR AA 5052 at at various solution treatments

The potentiodynamic polarization curves revealing the electrochemical corrosion performance of the ST-CR AA 5052 alloy in 3.5 wt.% NaCl solution is shown in figure 9. Often, the electrochemical corrosion performance is determined by the values of the corrosion potential (E_{corr}) and current (I_{corr}). Higher E_{corr} and lower I_{corr} often signify that the material is nobler. Table 2 shows the values of the E_{corr}, I_{corr} and corrosion rate obtained for the three ST-CR AA 5052 alloy. The summary of the corrosion performance of the cryorolled AA 5052 without ST and as-received metal, obtained under similar condition, from the previous work of the authors [2] were included in table 2.

As presented in table 2, it is apparent that all the ST-CR samples demonstrated better corrosion performance than the cryorolled sample without ST and much better than the as-received AA 5052 alloy because their I_{corr} are lower and E_{corr} are more positive (i.e. greater). This implies that the solution treatment caused significant improvement in corrosion resistance of the CR alloy. Generally, corrosion resistance or performance is related to the passive layers (i.e. thin-film protective oxide) formed on the material surface. The passive layers are formed during the passivation or oxidation process to protect the exposed material surface from corrosion damage. Hence, lesser material removal occurred due the passivation. According to Faradays’s laws of electrolysis, the mass of material removed is proportional to the amount of current passing through an electrolytic cell. Therefore, passivation at lower I_{corr} indicates better corrosion performance since severe material removal are found at higher I_{corr}. Passivation at lower I_{corr} quickly protect the exposed material surface from...
Figure 8. Fracture surface morphology of cryorolled AA 5052 solution treated at (a) 480 °C, (b) 510 °C, and (c) 540 °C.

Figure 9. Potentiodynamic polarization curve of cryorolled AA 5052 at varying solution treatment temperatures.

Table 2. Electrochemical corrosion test data for cryorolled 5052 at varying solution treatment temperatures.

| Samples                        | E_\text{corr} (V) | I_\text{corr} (\mu A) | Corrosion rate (mm/year) |
|--------------------------------|-------------------|------------------------|--------------------------|
| As received material           | −1.2602           | 12.00                  | 0.1971                   |
| CR sample without solution treatment | −1.2303       | 7.29                   | 0.1200                   |
| Solution treatment-CR at 480 °C | −1.1820           | 3.75                   | 0.0618                   |
| Solution treatment-CR at 510 °C | −1.1820           | 3.75                   | 0.0618                   |
| Solution treatment-CR at 540 °C | −1.1658           | 3.38                   | 0.0556                   |
| Solution treatment-CR at 540 °C | −1.1579           | 2.49                   | 0.0409                   |
severe corrosion damage [25]. Also, a more positive $E_{corr}$ is an indication that the material is nobler hence more formation of the passive layer during the corrosion process.

Comparing the three ST-CR aluminium alloy samples, the corrosion performance improved as the solution treatment temperature increased. Highly deformed grains containing a higher number of grain boundaries as expected on cryorolling are essential for the development of a high fraction of passive layer. Since this occurred in ST-CR aluminium alloy samples, better corrosion was expected. More so, better corrosion performance with increasing the ST temperature can be attributed to finer grain structure which improved as the temperature increased.

Figure 10 presents the SEM images of the cross-sectioned samples of the ST-CR AA 5052 alloy after the corrosion test. The irregularly shaped top layer on the samples were observed to be the corrosion product (i.e. the protective oxide layer) formed. The results of the EDX analysis conducted on the layers shows a significant presence of oxygen confirming that it is an oxide layer which is otherwise called the passive layer. The average thickness of the passive layer at ST temperatures of 480°C, 510°C and 540°C were found to be $5.3 \pm 0.4 \mu m$, $7.0 \pm 0.3 \mu m$ and $8.7 \pm 0.6 \mu m$ respectively. The oxide layer was not homogeneously distributed on the surface and open spaces of some oxide layers were present. This might be due to the effect of grain size distribution which is bimodal, consisting of a mixture of nanocrystalline and sub-microcrystalline grains and could contribute to the formation of non-uniform passive layers [26].

Although the passive layer on the surfaces was not uniformly formed, the result revealed that the average thickness of passive layers increases with the ST temperature. The cryorolled sample solution treated at 540 °C had more thickness and compact oxide layer compared to the other samples. Therefore, it can be concluded that the variation in the thickness of passive layers is related to the grain refinement, that permits different rates of oxide growth on the cryorolled surface. The large fractions of grain boundaries on the surface of cryorolled sample created a high number of active sites for the rapid formation of the continuous and protective passive film. The formation of high density nucleation sites for passive films led to a high fraction of passive layers and lowering of the corrosion rates [27]. Therefore, the UFG structures of the solution treated cryorolled samples, are more corrosion resistant, due to the conduction rate of the oxide layer on the surface which is being controlled by a high grain boundary density [28].

Figure 10. The oxide layer formed on the cryorolled AA 5052, solution treated at (a) 480 °C, (b) 510 °C, and (c) 540 °C.
4. Conclusions

The microstructure, mechanical and corrosion properties of cryorolled aluminium alloy 5052 pre-solution treated at various temperatures including 480 °C, 510 °C and 540 °C have been successfully investigated and compared with the as-received metal and cryorolled alloy without solution treatment. At the end of the work, the following conclusion were drawn:

(1) As compared to only cryorolling process, solution treatment followed by cryorolling produced AA 5052 with finer microstructure, higher hardness, yield strength, tensile strength and corrosion resistance. Both the cryorolled and solution treated cryorolled alloy showed better properties than the as-received metal.

(2) The hardness, yield strength, tensile strength and corrosion resistance of the cryorolled AA 5052 increased as the solution treatment temperature increased from 480 °C to 540 °C. The cryorolled AA 5052 solution treated at 540 °C shows improvements of about 15.1%, 7.1% and 4.8% in hardness, yield strength and tensile strength respectively, when compared with cryorolled alloy without solution treatment.

(3) Based on HRTEM analysis, the cryorolled alloy solution treated at 540 °C is characterised with high dislocations density and formation of bimodal microstructure consisting of submicron-sized and nano-sized grain structures. Also, EBSD analysis showed that the sample has significant variations in the orientation of neighbouring grains, a large number of submicron grains and a high fraction of low-angle grain boundaries. These are evidences of the formation of ultrafine-grained (UFG) structure.

(4) The smallest crystallite size and the highest value of lattice strain obtained were 24.88 nm and 3.86 × 10⁻⁵ respectively for cryorolled sample at solution treatment of 540 °C. The crystallite size reduced while the lattice strain increased as the solution temperature increased.

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ORCID iDs

T E Abioye @ https://orcid.org/0000-0003-2230-8770
A S Anasyida @ https://orcid.org/0000-0002-5655-7997

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