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# **Processing of nanostructured metallic matrix composites by a modified accumulative roll bonding method with structural and mechanical considerations**

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## **Abstract**

Particulate reinforced metallic matrix composites have attracted considerable attention due to their lightweight, high strength, high specific modulus, and good wear resistance. Al/B<sub>4</sub>C composite strips were produced in this work by a modified accumulative roll bonding process where the strips were rotated 90° around the normal direction between successive passes. Transmission electron microscopy and X-ray diffraction analyses reveal the development of nanostructures in the Al matrix after seven passes. It is found that the B<sub>4</sub>C reinforcement distribution in the matrix is improved by progression of the process. Additionally, the tensile yield strength and elongation of the processed materials are increased with the increase of passes.

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**Keywords:** metallic matrix composites; particle reinforced composites; nanostructures; aluminum; boron carbide; roll bonding; tensile properties

## 1. Introduction

Aluminum metallic matrix composites (MMCs) have attracted considerable attention due to their lightweight, high strength, high specific modulus, and good wear resistance. Boron carbide ( $B_4C$ ) is a common ceramic reinforcement in metallic matrix composites and presents considerable hardness, chemical resistance, and neutron absorbing properties [1-4]. Al/ $B_4C$  composites are used as a structural neutron absorber, armor plate materials, or a substrate material for computer hard disks [5-6]. There are several methods to manufacture this type of composites, such as powder metallurgy, squeeze casting, pressureless infiltration, and spray forming [7-9].

Accumulative roll bonding (ARB) has several advantages over other severe plastic deformation processes, including that (1) forming facilities with high load capacity and expensive dies are not needed, (2) the production efficiency is high, and (3) the content of materials to be produced is not limited. Recently, methods based on the accumulative roll bonding process were introduced to prepare metallic matrix composites, particularly for Al-SiC metallic matrix composites [10-11]. Additionally, a new forming method, namely cross-accumulative roll bonding (CARB) [12], which is based on the conventional accumulative roll bonding process, was successfully employed to process Al/ $B_4C$  metallic matrix composite sheets.

It was reported that the  $B_4C$  reinforcement distribution in Al/ $B_4C$  metallic matrix composites fabricated by accumulative roll bonding was improved by increasing the number of accumulative roll bonding passes, which was verified by hardness measurements where the

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standard deviation of hardness values decreased with the progression of accumulative roll bonding [13]. The homogeneity in terms of the B<sub>4</sub>C reinforcement distribution in the rolling direction-normal direction plane was in excess of the transverse direction-normal direction plane [14]. This paper aims to evaluate the structure and tensile properties of Al-10vol% B<sub>4</sub>C composites processed by the cross-accumulative roll bonding process as a function of the number of passes.

## 2. Experimental

Fully annealed 1050-aluminum strips and B<sub>4</sub>C powders with a mean particle size of 2 μm were used as the raw materials. The details of the cross-accumulative roll bonding process to produce Al-10vol% B<sub>4</sub>C composites were similar to Ref. [12]. To observe the dispersion of B<sub>4</sub>C particles in the matrix, the rolling direction-normal direction plane of the composites was polished to #3000 emery paper, finished with 1-μm diamond paste, and observed by an optical microscope. Here, the rolling direction was defined as the rolling direction of the first rolling pass. The optical figures were analyzed by the radial distribution function [13] to quantify the reinforcement distribution. In the radial distribution function, a circular disc of radius  $r$  is centered on the gravity center of a particle, and the radial distribution function ( $H(r)$ ) is determined as

$$H(r) = \frac{N_{ra}}{N_a} \quad (1)$$

where  $N_{ra}$  is the mean number of particles per unit area in a disc of radius  $r$ , and  $N_a$  is the mean number of particles per unit area over the whole sample. The degree of reinforcement

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clustering ( $A_H$ ) is estimated by the deviation of experimental radial distribution function curves from  $H(r)=1$  via the area  $A_H$  as described by

$$A_H = \int_{1\mu\text{m}}^{100\mu\text{m}} [H(r) - 1] dr \quad (2)$$

To determine the radial distribution function, a range of radii from  $r=1$  to  $100\mu\text{m}$  is considered.

The crystallite size of the Al matrix was determined by X-ray diffraction analysis (XRD, Philips Analytical PC-APD with a Cu  $K_\alpha$  radiation). Indeed, X-ray diffraction peaks are broadened when crystallites are small or the material contains lattice defects. The X-ray diffraction qualitative and quantitative analyses were conducted by X'Pert HighScore and MAUD programs, respectively. The MAUD software employs the Rietveld refinement to estimate the crystallite sizes by the Double-Voigt approach. A transmission electron microscope (TEM, Philips-FEG) was also used to observe the composite processed to seven passes.

Tensile test specimens oriented along the rolling direction were machined from the rolled sheets according to the 1/5 scale of JIS-No.5 specimen. The gauge length and width of the tensile test specimens were 10 and 5 mm, respectively. The tensile tests were carried out at ambient temperature and a nominal strain rate of  $8.3 \times 10^{-4} \text{ s}^{-1}$  using an Instron universal testing machine.

### **3. Results and discussion**

#### **3.1. Structure**

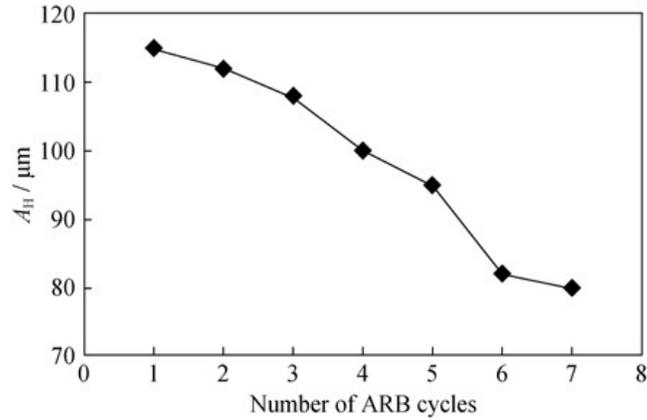
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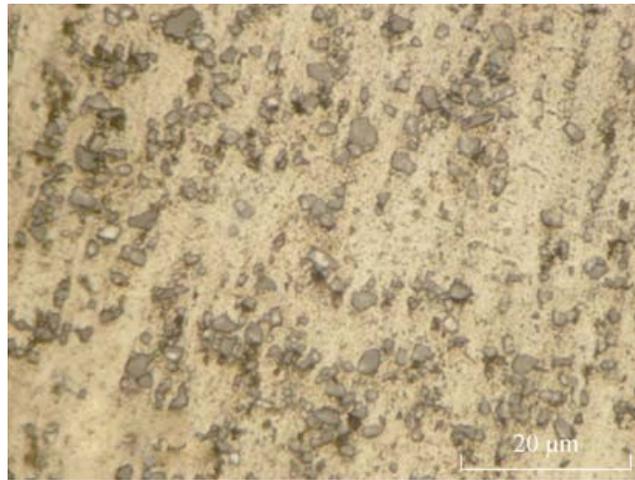
Fig. 1 depicts the degree of reinforcement clustering ( $A_H$ ), whose calculation procedure has been detailed in Ref. [13]. It is observed that the  $A_H$  values decrease with the increase of cross-accumulative roll bonding passes. Considering that larger  $A_H$  values are indicative of the increased particle inhomogeneity, it is inferred that, by increasing the number of cross-accumulative roll bonding passes, an improvement in the microstructure uniformity is obtained. The evolution of the B<sub>4</sub>C reinforcement distribution by progression of the accumulative roll bonding or cross-accumulative roll bonding processes can be explained in terms of the increase in the number of the Al and B<sub>4</sub>C layers, the metal extrusion through particle clusters, and the sheet elongation due to rolling [13-14]. Comparing the  $A_H$  values of the cross-accumulative roll bonding (Fig. 1) and accumulative roll bonding [13] processes, the uniformity in the rolling direction-normal direction plane of the sample accumulative roll bonded is better than that of the sample cross-accumulative roll bonded. It is due to the fact that, during the cross-accumulative roll bonding process, the transverse direction-normal direction and rolling direction-normal direction planes are alternatively interchanged, and the strain along the transverse direction in each pass is almost zero. Thus, the contribution of sheet elongation to the particle distribution in cross-accumulative roll bonding is half of that in the rolling direction-normal direction plane of the accumulative roll bonded samples where the sheet elongation along the rolling direction affects the evolution in all the passes. Typically, the optical micrograph of the composites produced by seven cross-accumulative roll bonding passes is given in Fig. 2, in which a relatively homogenous distribution of B<sub>4</sub>C particles into the Al matrix after seven passes can be observed. It is also notable that a structure without defects (porosities and cracks) has been developed as a result of the cross-accumulative roll bonding process to seven passes.

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**Fig. 1. Degree of clustering ( $A_H$ ) of the Al/B<sub>4</sub>C MMCs produced by the cross-accumulative roll bonding process.**



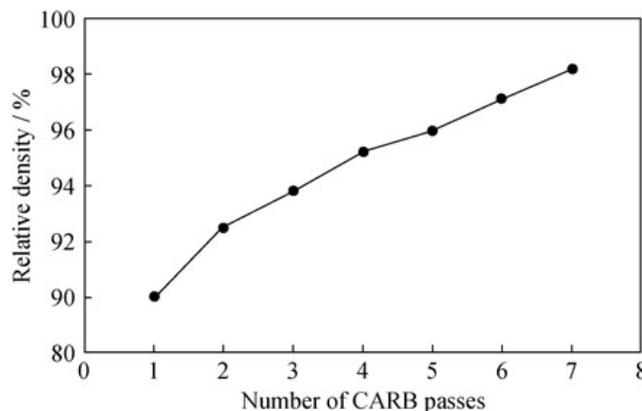
**Fig. 2. Optical micrograph of the composite produced by seven cross-accumulative roll bonding passes.**

The relative density of the processed composites is shown in Fig. 3. The increasing trend is attributed to the decrease in the size and number of discontinuities and porosities due to the improvement in the particle distribution and bonding quality. As focused above, the distribution of B<sub>4</sub>C particles is improved by progression of the accumulative roll bonding

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process. It means that the dissociation of clusters and agglomerations which include a considerable amount of discontinuities and porosities increases the relative density. It seems that the dense clusters have a more deleterious effect on the relative density compared to diffuse clusters. Because no matrix material is present between the particles of dense clusters and the level of their discontinuities is significant, while diffuse clusters include an amount of the matrix material between their particles. In addition, due to the increase in cross-accumulative roll bonding passes, a decrease in discontinuities of the interfaces (reinforcement/matrix and Al strip/Al strip) and accordingly the improvement of the bonding quality occur, which increases the relative density. As a result of the radial pressure and tangential shear stress of the rolling process having horizontal and vertical components, the matrix with a good formability is allowed to flow in different directions, reducing the porosities and discontinuities by progression of the process [15].



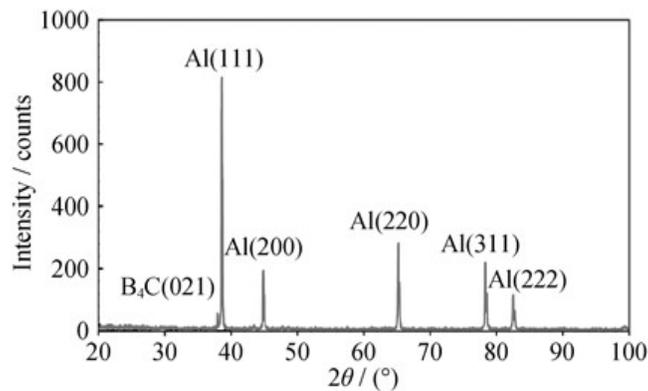
**Fig. 3. Relative density of the processed composites as a function of the number of cross-accumulative roll bonding pass.**

The X-ray diffraction pattern of the composite processed by seven cross-accumulative roll bonding passes is shown in Fig. 4, where the mean crystallite size is calculated to be 120 nm

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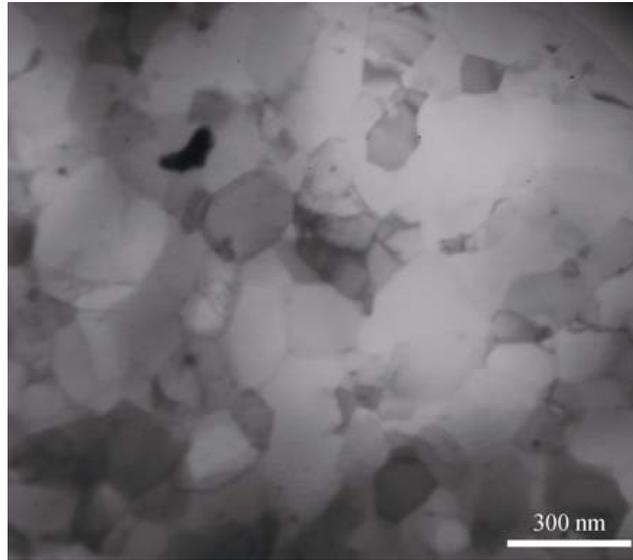
by the MAUD software, employing the Rietveld refinement and the Double-Voigt approach. In addition, the transmission electron microscope micrograph of the composite processed by seven cross-accumulative roll bonding passes is provided in Fig. 5. It can be seen that, in contrast to the conventional accumulative roll bonding process developing elongated grains, equiaxed nanostructured grains are present due to the fact that the strips are rotated  $90^\circ$  around the normal direction between the successive cross-accumulative roll bonding passes. It can be also concluded that the cross-accumulative roll bonding process to the seventh pass has successfully developed a nanostructured Al/B<sub>4</sub>C metallic matrix composite. Structural refinement can be explained in terms of grain subdivision at a submicron scale, where initial coarse grains are subdivided by deformation-induced high-angle grain boundaries [16-19]. Note that the crystallite size determined from X-ray line broadening is smaller than the related grains observed by the transmission electron microscope due to the subdivision of grains into substructures.



**Fig. 4. X-ray diffraction pattern of the composite processed by seven cross-accumulative roll bonding passes.**

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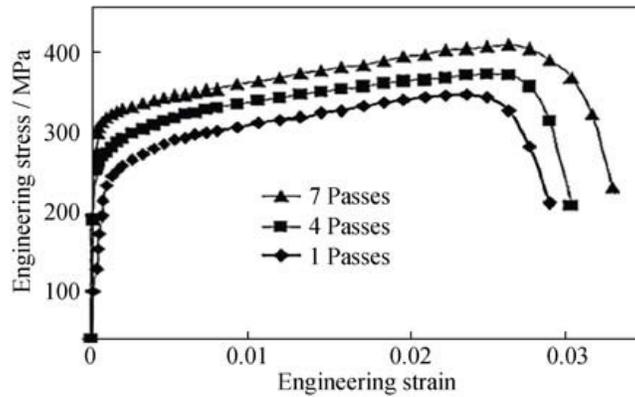
**Fig. 5. Transmission electron microscopy micrograph of the composite processed to seven passes.**

### **3.2. Tensile properties**

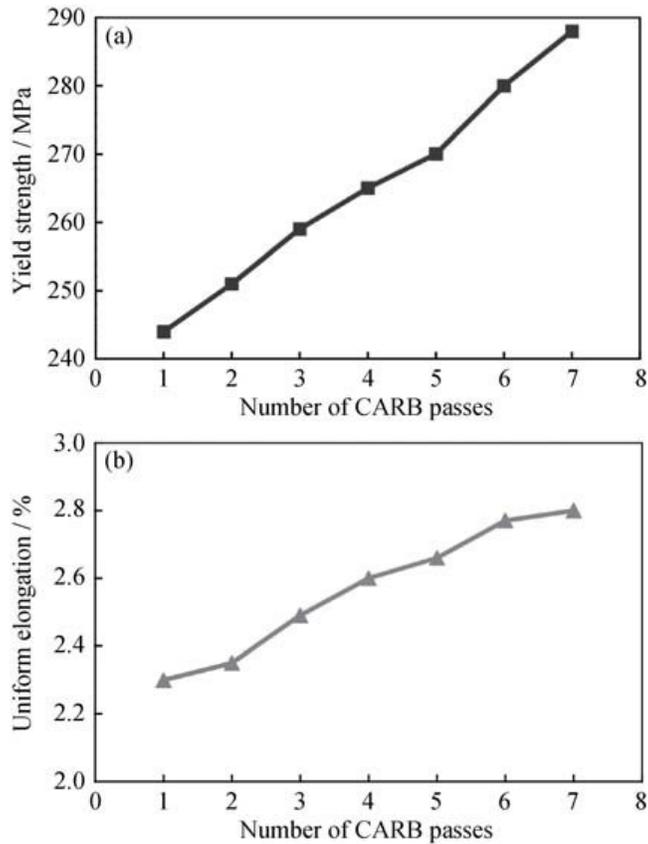
Figs. 6 and 7 represent the tensile stress-strain curves and the results of tensile tests (yield strength and uniform elongation) of the produced composites, respectively. It can be seen that the yield stress and elongation increase by progression of the process. It was reported that [15], by increasing accumulative roll bonding passes to seven passes, the tensile elongation of Al-10 vol% SiC metallic matrix composites decreased, but it was improved when the number of accumulative roll bonding passes exceeded nine, where the total SiC reinforcements were inserted into the composite step by step to five accumulative roll bonding passes. However, in the current study, the cross-accumulative roll bonding process was defined to start when all the B<sub>4</sub>C particles were added to the specimens. The observed tensile behavior can be explained from several viewpoints as follows.

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**Fig. 6. Stress-strain curve of the composites prepared by the cross-accumulative roll bonding process.**



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**Fig. 7. Yield strength (a) and uniform elongation (b) of the composites as a function of cross-accumulative roll bonding passes.**

- (1) Bonding quality between Al layers (interlayer bond strength). In the first cross-accumulative roll bonding passes, there is no adequate bonding between the Al layers, and the interfaces include a considerable amount of discontinuities and porosities. These defects develop a significant stress concentration and triaxial stresses within the matrix, thereby decreasing the yield stress and elongation. By progression of the cross-accumulative roll bonding process, a better bonding between the Al layers and accordingly an improvement in the mechanical properties are obtained, which is in accordance with Refs. [10-11]. The decrease of discontinuities and porosities by increasing cross-accumulative roll bonding passes is supported by the evolution of the relative density in Fig. 3. About the effect of the particle presence on the interlayer bond strength, there were antitheses between obtained results by different researchers. Lu *et al.* [20] found that nanoparticles between matrix layers enhanced the interlayer bond strength, which depended on the hardness of introduced particles. Because the particles between the sheets induce a stress concentration during rolling, thereby increasing the cracking of surface layers on the top of surfaces to be joined. In contrast, it was reported that the particulate reinforcements slightly deteriorated the interlayer bond strength during accumulative roll bonding, since they reduced the metal-to-metal contact area of two sheets to be roll bonded [21].
- (2) Strain hardening (dislocation strengthening). In the initial passes, the dislocation density progressively increases as a result of plastic deformation, and the related strain

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hardening plays a main role in strengthening [22-26], reducing the tensile elongation, which is in good agreement with Refs. [10, 24-25].

(3) Grain boundary strengthening. The gradual evolution of grain structure and the formation of ultrafine grains and nanostructured subgrains by progression of the process (Figs. 4 and 5) significantly increase the strength, particularly in final passes [22-26]. Tsuji *et al.* [26] pointed out that strain hardening due to the accumulative roll bonding process difficultly occurred when the grain size was below 1  $\mu\text{m}$ . That is, in high accumulative roll bonding passes, the role of structural refinement in strengthening prevails over that of work hardening. Concerning the effect of reinforcement on the grain refinement during the accumulative roll bonding process, an accelerated refinement in the presence of particles was reported due to additional local straining around the embedded particles [27], as combined with the redundant shear strain at the sheet surfaces introduced by surface forces during roll bonding [17]. By increasing the dislocation density due to the presence of the particles, a severe structural refinement is obtained, increasing the strength based on the grain boundary strengthening mechanism. Furthermore, the retardation of grain boundary movement owing to the existence of the particles and the possibility of matrix recrystallization *via* particle-stimulated nucleation provide a smaller grain size in the matrix of composites compared to the unreinforced alloys [28].

(4) Bonding quality of Al/B<sub>4</sub>C interfaces. The Al/B<sub>4</sub>C interfaces are preferred sites for the initiation and propagation of cracks during plastic deformation. In initial passes, there is a considerable level of discontinuities and porosities in the Al/B<sub>4</sub>C interfaces. By increasing passes, the discontinuities and porosities decrease (Fig. 3), and accordingly the interface strength is enhanced, due to the good formability of the matrix and the act of radial pressure

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and tangential shear stress [29], leading to the enhancement of yield strength and elongation. It has been also found that, when the amount of deformation exceeds 90%, all these porosities can be eliminated [30].

(5) Reinforcement distribution. The reinforcement distribution is improved by increasing passes as shown in Fig. 1. Particle clusters, especially dense clusters, produce a considerable amount of discontinuities and porosities and consequently reduce the yield stress and elongation [31-33]. The dissociation of clusters is one of the important features of the improvement in the reinforcement distribution and dictates an enhancement in the yield stress and elongation. Because the distance of Al/B<sub>4</sub>C interfaces as the preferred crack or decohesion nucleation sites increases, and the crack coalescence occurs later. In addition, in initial passes, the material has a layered structure, in which the layers of B<sub>4</sub>C powders keep the soft metal layers apart. Obtaining the uniform distribution of the reinforcement due to the increase in passes, the particle-free zones with low strength are shrunk, and the strengthening effect of the reinforcement is activated.

(6) Reinforcing effect of B<sub>4</sub>C particles. The reinforcing B<sub>4</sub>C particles enhance the critical stress of dislocation glide and result in the creation of additional dislocations around the particles. These dislocations are generated at the particle-matrix interface to accommodate the strain incompatibility between two phases [10]. Thus, the interaction of dislocations increases and the mobility of dislocations decreases, leading to an increase in strength and a decrease in ductility. In addition, the B<sub>4</sub>C particles act as barriers to dislocation movement, contributing to additional strengthening. That is, the presence of the B<sub>4</sub>C particles enhances work hardening during plastic deformation.

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(7) Mismatch between the coefficients of thermal expansion. During plastic deformation, an increase in sample temperature has been pointed out [34]. Because of the difference in the coefficients of thermal expansion between Al and B<sub>4</sub>C, dislocations are created at Al/B<sub>4</sub>C interfaces after any passes. These newly formed dislocations develop multidirectional thermal stresses in the structure, thereby increasing the strength and decreasing the tensile elongation. It can be concluded that a number of the aforementioned factors essentially result from the cross-accumulative roll bonding process and some are a consequence of the presence of B<sub>4</sub>C reinforcement. As noted, all the seven factors support the observed increase in yield stress of the composite by progression of the process. Nonetheless, for the studied composites, the increase in tensile elongation suggests that the contributions of the improvement in the interlayer bond strength, the quality of Al/B<sub>4</sub>C interfaces, and the reinforcement distribution prevail over those of the other deleterious factors.

#### **4. Conclusions**

- (1) Metallography studies by the radial distribution function suggest an improvement in the B<sub>4</sub>C reinforcement distribution by progression of cross-accumulative roll bonding passes.
- (2) The transmission electron microscopy and X-ray diffraction peak profile analyses reveal the development of nanostructures in the Al matrix after seven cross-accumulative roll bonding passes.
- (3) An increase is found in the tensile yield strength and elongation of the composites by increasing the cross-accumulative roll bonding passes.

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