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Influence of Heat-Treatment on the Adhesive Strength between a Micro-Sized Bonded Component and a Silicon Substrate under Bend and Shear Loading Conditions

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Abstract Adhesive bend and shear tests of micro-sized bonded component have been performed to clarify the relationship between effects of heat-treatment on the adhesive strength and the bonded specimen shape using Weibull analysis. Multiple micro-sized SU-8 columns with four different diameters were fabricated on a Si substrate under the same fabrication condition. Heat-treatment can improve both of the adhesive bend and shear strength. The improvement rate of the adhesive shear strength is much larger than that of the adhesive bend strength, because the residual stress, which must change by heat-treatment, should effect more strongly on the shear loading. In case of bend type test, the adhesive bend strength in the smaller diameters (50 and 75 μm) widely vary, because the critical size of the natural defect (micro-crack) should vary more widely in the smaller diameters. In contrast, in case of shear type test, the adhesive shear strengths in each diameter of the columns little vary. This suggests that the size of the natural defects may not strongly influence on the adhesive shear strength. All the result suggests that both of the adhesive bend and shear strengths should be complicatedly affected by heat-treatment and the bonded columnar diameter.

Keywords: SU-8, Adhesive Bend Strength, Adhesive Shear Strength, Materials Testing Machine for Micromaterials, Weibull Distribution

1. Introduction

In the last few decades, chip making technology has been advancing rapidly with improvements in microfabrication techniques[1]. Most of the techniques are based on photo-lithography, which is a processing technique to fabricate fine patterns of photoresist film on a wafer for two-dimensional fine curcuits in integrated circuits(ICs). The technique has been modified to fabricate three-dimensional micro mechanical elements with high aspect ratio in micro-electro mechanical systems(MEMS); for example, surface micromachining[2], reactive ion etching(RIE)[3], anisotropic etching[4], LIGA process[5,6], Bosch process[7], etc. MEMS devices are built by laminating fine patterns using these techniques, thus, the devices include a large number of interfaces between dissimilar materials, e.g. combination of silicon, metals, ceramics, and/or polymers. There are many sources of stress concentration at the interface, for example, natural defects, residual stress caused by difference of thermal expansion coefficients, difference of elastic moduli etc. As a result, fracture sometimes occurs near the interface, thus, the delamination is one of the most serious problem in MEMS. However, there are many factors to complicatedly effect the delamination during fabrication and/or in use. It

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is necessary to quantitatively evaluate the adhesive strength between a micro-sized component and a substrate for the purpose of clarifying the relationship between effective factors and the adhesive strength.

In our precious study, quantitative adhesive testing methods have been developed for micro-sized bonded components on a substrate using columnar shape of the micro-sized bonded component (see Fig. 1)[8,9]. The columnar shape has been adopted as a bonded test piece in this method, because it is easy to fabricate multiple test pieces on a substrate under the same process condition. Multiple test pieces with the same dimensions, which have been fabricated under the same processing condition, should be prepared in order to statistically alanyze obtained experimental data of the adhesive strength; because the data sometimes vary widly. Two loading types are available for the adhesive test method, bend and shear loading types, as shown in Figs. 1a) and b) respectively[10].

We have been studying the adhesive strength between a micro component made of epoxy type photoresist, SU-8 and Si substrate using this method, focused on processing conditions, for example, exposure dose in lithography and heat-treatment[11]. It was clarified that heat-treatment can significantly improve the adhesive bend strength between a micro-sized SU-8 column and silicon substrate[11]. The influence

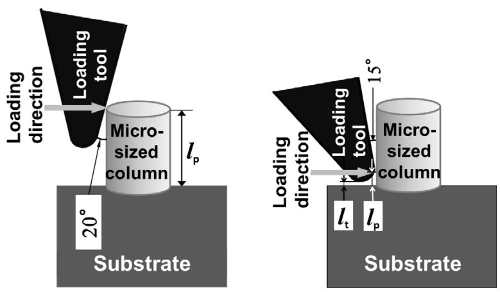


Fig. 1 Adhesive strength tests for micro-sized bonded components on a substrate

rate by heat-treatment seems to change by the columnar height of a bonded SU-8 with the same diameter. However, it is unclear that which actually effects on the influence rate, columnar height (i.e. SU-8 film thickness), aspect ratio of the column, and/or another factors.

In this study, adhesive strength between a micro-sized SU-8 column and Si substrate has been quantitatively evaluated for the purpose of clarifying the relationship between effects of heat-treatment on the adhesive strength and bonded specimen dimension. Both bend and shear type tests were performed to clear how the heat-treatment effects on the adhesive strength.

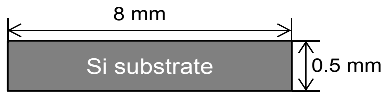
Mechanical properties of SU-8 should be changed by film thickness under the same processing condition, thus, four different diameters of SU-8 columns with the same columnar length (film thickness) were fabricated on a Si substrate under the same processing condition in this study. Multiple test pieces with each columnar diameter were prepared to statistically evaluate the adhesive strength by Weibull distribution. All the results should make clear the effects of the heat-treatment on the adhesive strength in micro-sized bonded SU-8.

2. Experimental

2.1 Materials and Fabrication Details

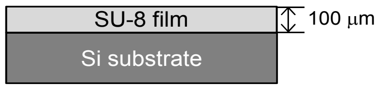
Materials used in this study are an epoxy type photoresist, SU-8 and a Si substrate of 0.5 mm thickness. SU-8 can be easily fabricated three dimensional micro components with several tens microns in thickness using UV photo-lithographic technique, although it is difficult for common photoresist to fabricate such a three-dimensional fine patterns[12]. SU-8 is utilized as permanent materials for micro fluidics, wave guide, soft cantilever, etc. Thus, the resist was selected as the test piece in this study to study the adhesive strength of SU-8 for safe design.

Film type of SU-8 with 50 μm in thickness, SU-8 3000 film (made by Nippon Kayaku Co., Ltd.) was selected, because liquid type of SU-8 for thick film is too viscous to precisely control the film thickness. Fig. 2 shows fabrication procedure of adhesive columnar test pieces in this study. A Si wafer was cut into approximately 8 mm square and it was cleaned up using alkali cleaning solution, distilled water, acetone, and ethanol sequentially (see Fig. 2(a)). Two sheets of SU-8 film were laminated on a Si substrate using a rubber roller at 333 K on a hot plate (see Fig. 2 (b)). After the lamination, the SU-8 films were heat-treated at 353 K for 8 min for smoothing of the film surface and the interface (see Fig. 2(c)). Micro-sized SU-8 columns with four different diameters (50, 75, 100, and 125 μm in diameters) were fabricated on the Si using photolithographic technique (see Fig. 2(d) and Fig. 3). Multiple columnar test pieces (approximately 40 pieces) with each diameter were prepared on the Si substrate as shown in the right figure in Fig. 3. Table 1 shows the photolithography condition in this study.

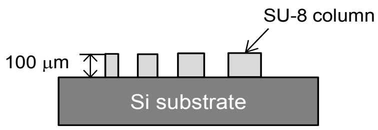


(a) Substrate preparation (cutting and cleaning)

(b) Resist film lamination (two sheets of SU-8 film were laminated at 333 K)



(c) Heat treatment (at 353 K for 8 min)



(d) Microfabrication by photolithography

Fig. 2 Microfabrication procedure of adhesive columnar test pieces

After the fabrication, both columnar diameter and length in each SU-8 column were measured using a confocal laser scanning microscope, 1LM21 (CLSM, made by Lasertec Corp.) with a horizontal resolution of 0.30 μm, horizontal accuracy of 0.03 μm, and a vertical accuracy of 0.03 μm. Table 2 shows mean measured values of each target columnar dimensions. The dimensional error of each SU-8 columns with the same diameter is within several microns (approximately 3%).

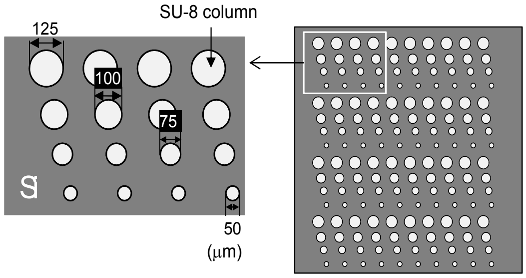


Fig. 3 A schematic diagram of columnar test pieces with four different diameters on a Si substrate

Table 1 Fabrication condition (photolithography)

Exposure dose	600 mJ/cm^2 by UV light
Post exposure bake	1) 338 K for 3 min 2) 368 K for 6 min 3) Relaxation for 15 min
Development	Developer: SU-8 developer Temperature: 298 K Developing time: 10 min
Rinse	IPA

Table 2 Dimensions of micro-sized columnar test piece (μm)

Target columnar dimensions		Mean measured values	
Diameter (D)	Length (l)	Diameter (D)	Length (l)
50	100	51.3	101.9
75		75.9	101.5
100		100.8	101.9
125		125.9	101.7

2.2 Adhesive Strength Test Methods

In case of bend type test, the bending force is applied to the end of the columnar test piece using a loading tool, which is 20° tilted against the lateral face of the test piece as shown in Fig. 1(a).

Adhesive bend strength (σ_a) is calculated by the following equation;

$$\sigma_a = \frac{M_a}{Z} = \frac{32 F_{Ba} l_p}{\pi D^3} \tag{1}$$

where M_a denotes the bend moment at delamination, Z is the section modulus of a columnar test piece, F_{Ba} is the bending force at delamination, D is a diameter of a columnar test piece, and l_p denotes the loading distance (see Fig. 1(a)). The columnar length is defined as l_p in this study.

On the other hand, in case of measuring adhesive shear strength; the line load should be applied to the columnar test piece using a loading tool, which is parallel to the lateral face of the test piece. However, it is difficult to precisely align between the micro-sized test piece and loading tool. Instead, a point load was applied at the vicinity of the columnar root using a loading tool with an approximately slightly slanting its apex as shown in Fig. 1(b). In such a case, the effect of bend stress at the columnar root can be minimized and the delamination must mainly occur by shear loading[8].

Adhesive shear strength (τ_a) is calculated by the following equation;

$$\tau_a = \frac{F_{Sa}}{A} = \frac{4 F_{Sa}}{\pi D^2} \tag{2}$$

where A denotes the adhesive joint area and F_{Sa} is the shear force at delamination.

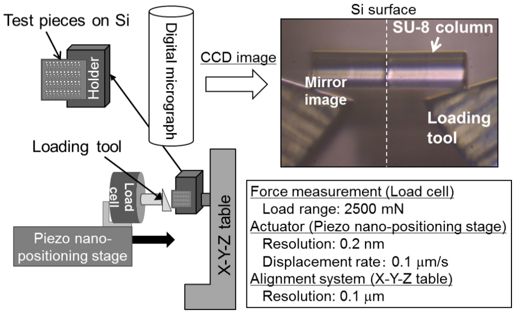


Fig. 4 Adhesive strength measurement system between a microsized columnar test piece and substrate

2.3 Test Equipment and Test Condition

Fig. 4 shows the adhesive strength measurement system in this study. A material testing machine for micromaterials, which has been developed in our previous study, was used for the adhesive tests[13]. The machine has a piezo type of precise x stage with the maximum displacement of 38 μm (piezo nano-positioning stage (P753.31C, made by Physik Instrumente (PI)), which can apply precise displacement. The applied force can be measured by a precise load cell with load range of 2500 mN (Model 31, made by Honeywell). A X-Y-Z tables and a digital micrograph with CCD camera in the machine were used for alignment between a test piece and a loading tool. A CCD image in Fig. 3 shows example of the alignment in bend type test.

Both adhesive bend and shear tests were performed at a displacement rate of 0.1 $\mu m/s$ at room temperature. Firstly, non-heat-treated SU-8 columns were tested under bend and shear loading for obtaining the reference data. After then, the test pieces were heat-treated at 423 K for 30 min.

Approximately ten pieces of SU-8 columns with each diameter were tested in each test condition for the statistical data analysis by Weibull distribution.

2.4 Data Aalysis

Weibull existence probability(S) at adhesive bend strength(σ_a) or adhesive shear strength(τ_a) was calculated as;

$$S = \exp(-\sigma_a^m) \quad (3)$$

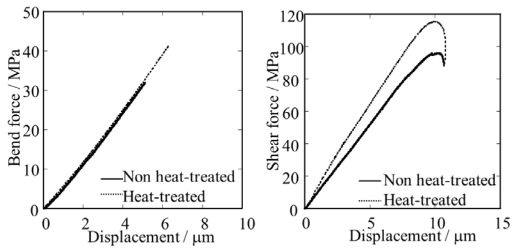
$$S = \exp(-\tau_a^m) \quad (4)$$

$$S_j = 1 - \frac{j}{N+1} \quad (5)$$

where m denotes the Weibull modulus, N is sample number, S_j is the mean value of S for the j-th sample taken from N, and j-th denotes weak order. m value shows the variation in the experimental data of adhesive bend or shear strength. For, example, smaller m shows that the obtained data are more widely scattered.

3. Results and Discussion

3.1 Force-Displacement Curves in Bend and Shear Type Tests



(a) Bend type tests (b) Shear type tests

Fig. 5 Typical force-displacement curves in bend and shear type tests using SU-8 columnar test pieces 100 μm in columnar diameter and 100 μm in columnar length

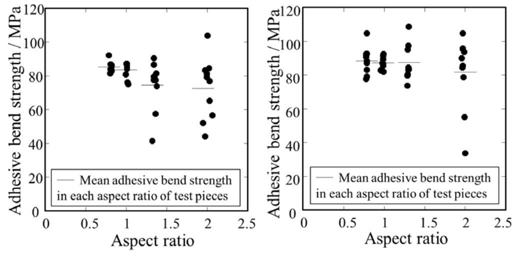
Fig. 5 shows typical force-displacement curves, which were obtained from the adhesive tests using SU-8 columnar test pieces with and without heat-treatment. In case of bend type test, applied bending force linearly increased until the maximum force, and then, the force suddenly dropped in a brittle manner (see Fig. 5(a)). This shows that the delamination should occur at the maximum force, thus, the maximum force is defined as F_{Ba} in this study. In addition, the slope of the curves is almost the same in both of non-heat-treated and heat treated SU-8 test pieces, although the maximum force became larger by heat-treatment. It seemed that the heat-treatment little affected Young's modulus of the SU-8, in spite of improvement of adhesive strength by heat-treatment in this study.

In case of shear type test, applied shear force linearly increased until the maximum force, then, the force was in a gradual decline for only a brief moment. After then, the applied shear force suddenly dropped by the delamination (see Fig. 5(b)). It is suggested that crack propagation for the delamination should slowly start at the maximum force firstly. Therefore, the maximum force is defined as F_{Sa} in this study. In addition, both the slope of the curve and the maximum shear force became larger by heat-treatment. It seems shear modulus of SU-8 changes by heat-treatment in shear type test, which is inconsistent in the fact that the Young's modulus little changes in bend type test. This suggests another factor(s) to change the slope of the load-displacement curve by heat-treatment, for example, residual stress at the interface.

3.2 Effects of Heat-Treatment on the Adhesive Strength

Fig. 6 shows the relationship between the adhesive bend strength and aspect ratio of the columnar test piece (columnar length / columnar diameter). All columnar test pieces have the same columnar length in this study, thus, higher aspect ratio shows smaller columnar diameter.

In case of non-treated test pieces, the adhesive bend strengths in aspect ratio of 0.8 and 1.0 (the lower aspect ratios) tend to be slightly higher than those in aspect ratios of 1.3



(a) (b)

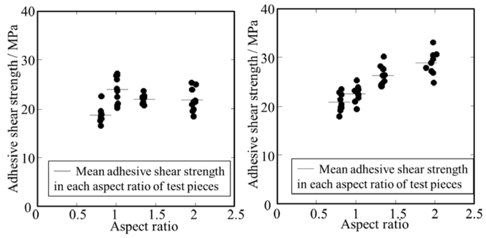
Fig. 6 Adhesive bend strength between a SU-8 columnar test piece and silicon substrate vs. the aspect ratio using the test pieces with (a) Non-heat-treatment and (b) Heat-treatment

Table 3 Mean adhesive bend strength in each SU-8 column with 100 μm in height

SU-8 column		Adhesive bend strength		
Diameter /μm	Aspect ratio	Non-treatment /MPa	Heat-treatment /MPa	percentage change
50	2.0	72.8	81.7	12.2%
75	1.3	74.7	87.2	16.7%
100	1.0	83.7	86.9	3.8%
125	0.8	85.5	88.4	3.4%

and 2.0 (the higher aspect ratios) (Fig. 6(a)). In contrast, the adhesive bend strengths seem almost constant in all aspect ratios in heat-treated test pieces (Fig. 6(b)). However, all experimental data tend to widely vary; thus, it is unclear whether the adhesive bend strength changes by heat-treatment in these graphs of Fig. 6.

Mean adhesive bend strength in each diameter of the test piece is shown in Table 3. The adhesive bend strength in the non-heat-treated test pieces became higher by heat-treatment, although the percentage change of the improvement seems different by aspect ratios of the test pieces. The adhesive bend strengths in the higher aspect ratio are more than ten percent improved by heat-treatment. In contrast, the improvement rate is less than 4% in the lower aspect ratios. As a result, regardless of the aspect ratio, mean adhesive bend strength is almost constant in the heat-treated test pieces.



(a) (b)

Fig. 7 Adhesive shear strength between a SU-8 columnar test piece and silicon substrate vs. the aspect ratio using the test pieces with (a) Non-heat-treatment and (b) Heat-treatment

Table 4 Mean adhesive shear strength in each SU-8 column with 100 μm in height

SU-8 column		Adhesive shear strength		
Diameter / μm	Aspect ratio	Non-treatment /MPa	Heat-treatment /MPa	percentage change
50	2.0	21.8	28.9	32.6%
75	1.3	21.8	26.3	20.6%
100	1.0	23.9	22.6	-5.4%
125	0.8	18.6	20.9	12.4%

Fig. 7 shows the relationship between the adhesive shear strength and the aspect ratio. In case of non-heat-treated test pieces, the adhesive shear strength is almost constant between each aspect ratio. In contrast, the adhesive shear strength seems to increase with increasing the aspect ratio in the heat-treated test pieces.

Mean adhesive shear strength in each diameter of the test piece is shown in Table 4. The adhesive shear strength tends to be improved by heat-treatment; especially the strength with higher aspect ratios became approximately 20-30% higher than the lower aspect ratios.

The above results show that heat-treatment can improves both of the adhesive bend and shear strength. In addition, the improvement rate of adhesive shear strength is much larger than that of adhesive bend strength. This suggests a factor(s) to change by heat-treatment, which is more effective against the shear loading, for example, residual stress. Residual stress at the interface must be generated at the interface, which is perpendicular to the shear loading direction; thus, the residual stress should more strongly effects the shear loading. In addition, the influence rate of the adhesive strength tends to increase with increasing the aspect ratio. It is difficult to explain the fact using only the effects of residual stress in this study. The further studies are needed.

3.3 Weibull Analysis

Fig. 8 and 9 show Weibull existence probability plots for the adhesive bend and shear strengths in each columnar diameter. A straight line is fitted to the Weibull plots in each test piece (non-heat-treated and heat-treated specimen) to define m value (Weibull modulus). Table 5 shows m value in each diameter of the test piece.

In case of bend type test (Fig. 8), m value is similar between non-heat-treated and heat-treated test pieces in each columnar diameter. In addition, m value is much smaller in the test pieces with 50 and 75 diameters (the higher aspect ratio) than the others. In other words, experimental data of the adhesive bend strength vary more widely in the higher aspect ratio (i.e. the smaller columnar diameter). It is suggested that there is an effective factor(s) to widely vary in the smaller columnar diameter. The effective factors on the data distribution should be natural defects (or natural crack) at the interface between a bonded SU-8 column and Si substrate, because SU-8 components sometimes include micro-cracks during fabrication[13].

The difference of m value between the smaller diameters and the larger diameters should indicate the difference of the critical micro-crack size, which causes the delamination. In case of the smaller diameters, critical size of the micro-crack at the edge of the interface should widely vary in each test piece; as a result, the adhesive bend strength should vary widely. In contrast, in case of the larger diameters, the critical micro-crack size should little vary. This trend does not change by heat-treatment.

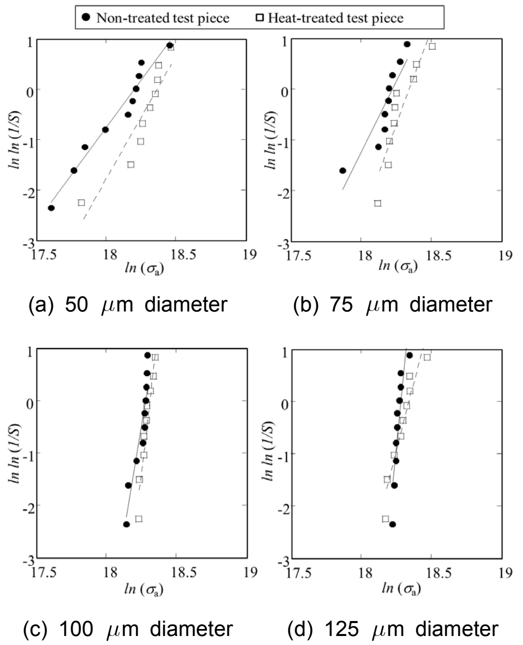
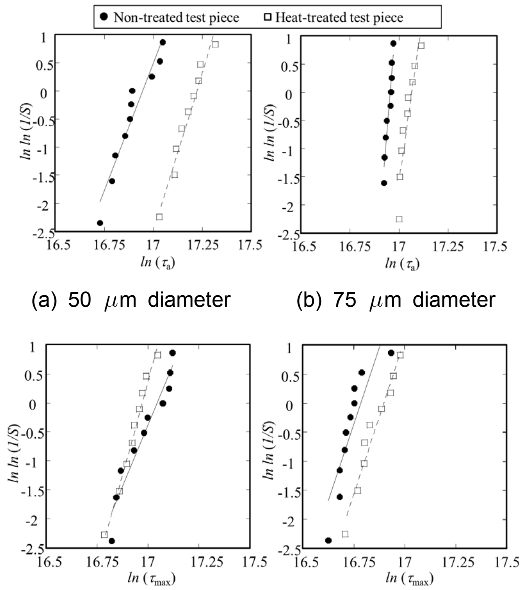


Fig. 8 Weibull existence probability plots for adhesive bend strength in each columnar diameter of the test piece. Each slope of the fitted line shows “*m*” value

Table 5 “*m* value” in each dimensions of SU-8 columns

SU-8 column		*m* value			
		In bend type test		In shear type test	
Diameter / μm	Aspect ratio	Non-treatment	Heat-treatment	Non-treatment	Heat-treatment
50	2.0	3.8	4.9	9.1	11.4
75	1.3	5.6	7.5	12.2	21.6
100	1.0	16.9	23.4	8.4	12.6
125	0.8	26.3	10.3	10.6	10.7

In case of shear type tests, most m values are large, which shows that data of the adhesive shear strength little vary. This suggests that the effective factor(s) for adhesive bend strength, which may be the critical micro-crack size,



(a) 50 μm diameter (b) 75 μm diameter

(c) 100 μm diameter (d) 125 μm diameter

Fig. 9 Weibull existence probability plots for adhesive shear strength in each columnar diameter of the test piece. Each slope of the fitted line shows "*m* value"

should not strongly affect the adhesive shear strength.

All the results suggest that the effective factor(s) on the delamination should be different between bend and shear loading conditions.

4. Conclusions

Adhesive bend and shear strength between a micro-sized SU-8 and Si substrate has been evaluated to clarify the relationship between the influence rates of heat-treatment on the strengths and the dimension of bonded SU-8 columns. The results and discussions are concluded as follows;

1) Heat-treatment can improve both of the adhesive bend and shear strength. The improvement rate of the adhesive shear strength is much larger than that of adhesive bend strength. It is suggested that there is a factor(s) to change by heat-treatment, which is more effective against the shear loading, for example, residual stress.
2) The improvement rate of the adhesive strength tends to increase with increasing the aspect ratio.
3) In case of the delamination by bend loading, the adhesive bend strengths in the smaller columnar diameters more widely vary than those in the larger diameters. It should be reflected in the difference of the critical micro-crack size at the interfaces in each diameter of the test piece.
4) In case of the delamination by shear loading, the adhesive shear strengths in each columnar diameter little vary. It is suggested that the distribution of the natural crack size little effects on the adhesive strength in this study.

All the result suggests that both of the adhesive bend and shear strengths should be complicatedly affected by heat-treatment and the bonded columnar diameter.

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