Scratch Hardness of Stone Models – Measuring Conditions

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Received January 30, 2004/Accepted August 31, 2004

This study investigated the effects of scratch load, scratch speed, and storage period of specimen on the scratch hardness of stone models. Types 4 and 5 stone specimens were made from vinyl silicone rubber and alginate impressions respectively. After specimens were stored in a desiccator with silica gel at room temperature for 24 hours or 1 week, scratches were created on the surface with three different scratch loads (1N, 2N, and 3N) and two different scratch speeds (50 and 100 mm/min). Then, the depths of scratches were measured.

Scratch depth of stone specimens increased with increasing scratch load. Scratch depths of Type 4 stone specimens after 1-week storage were smaller than those after 24-hour storage at a speed of 100 mm/min. However, the effect of scratch speed on scratch depth did not follow a specific trend.

Key words: Stone model, Surface hardness, Scratch test

INTRODUCTION

For strict infection control in dentistry, it is absolutely necessary to disinfect impressions to prevent transmission of infectious diseases to dental personnel^{1–3}. It has been reported that depending on the disinfection method and type of impression material, disinfection of impressions can have varying effects on the resultant stone models in terms of surface roughness, detail reproducibility, dimensional stability, and deformation^{4–8}. However, only a few reports focused on investigating the effects on the surface hardness of stone models obtained from disinfected impressions^{9,10}. Stone models are expected to be sufficiently hard to resist marginal fracture or abrasion during carving. However, disinfection of impressions has been suspected to reduce surface hardness of stone models due to effects arising from water absorption, impression dissolution, or remaining disinfectant solution on impression surface. Therefore, a hardness evaluation study would provide valuable information on how to develop a disinfection method for impressions that does not compromise the reproducibility of stone models.

The Knoop hardness test, a test based on resistance to indentation, has been used to evaluate the hardness of stone models^{10–13}. However, measurement can be difficult because indentation produced on the stone model surface is not well defined. It is particularly difficult to observe indentation produced on a deteriorated surface of stone model which is disinfected or obtained from disinfected impression. Hardness test based on the ability to resist

scratching in stone models exists and utilizes depth measurements at several locations along a groove created by specified load¹⁴. The scratch test is considered more suitable for determining surface hardness of materials that are not amenable to receiving clear indentation, such as stone models obtained from disinfected impressions. In the present study, we investigated the effects of scratch load, scratch speed, and storage period of specimen on the scratch hardness of stone models.

MATERIALS AND METHODS

Materials listed in Table 1 were used according to manufacturers' instructions. An alginate impression material (AFIII) was mixed for 15 seconds using an automatic alginate impression material mixer (Rakuneru, GC, Tokyo, Japan). The vinyl silicone rubber impression material (EMF) was automatically mixed using a dispenser designed for this purpose. Type 4 gypsum product (NF) and Type 5 gypsum product (NP), classified in accordance with the International Standard, were selected for EMF and AFIII impressions respectively. Both NF and NP were first mixed by hand for 45 seconds and then vibrated for 30 seconds.

Preparation of stone specimens

Stone specimens were produced using the equipment shown in Fig. 1. Procedures were performed as follows: 1. The mixed impression material was filled into a perforated ring tray with a base of 30-mm internal diameter, 5-mm depth, and 2-mm thickness.

Table 1 Materials used

Code	Brand name	Manufacturer	Lot No.	W/P (ml/g)
<u>Alginate impression material</u>				
AFIII	Aroma Fine DFIII	GC	0102213	2.38
<u>Vinyl silicone rubber impression material</u>				
EMF	Examix Fine Regular type	GC	0304101	—
<u>Dental stone</u>				
NP	New Plastone	GC	0211111	0.24
NF	New Fujirock	GC	0301101	0.20

Fig. 1 Tray (left) and rubber mold (right) used for making specimens.

2. A clean, flat plastic plate was placed on top of the mixture and held to make an impression of the plastic plate. 3. The plastic plate was removed either 7 minutes after the start of alginate mixing to make the AFIII impression, or 15 minutes after the start of silicone rubber injection to make the EMF impression. 4. The mixed stone was poured onto the surface of the impression mounted in a circular rubber mold with a height of 15 mm, and was allowed to set at room temperature. 5. One hour after stone pouring, the stone specimen held by the rubber mold was removed from the impression. The disc-shaped stone specimen separated from the rubber mold was kept in a desiccator with silica gel at room temperature for 24 hours or 1 week before the scratch hardness test.

Scratch hardness test

The scratch test was performed using a surface property tester (Heidon-14, Shinto-Kagaku, Tokyo, Japan) and a profilometer (Surfcom 554A, Tokyo Seimitsu, Tokyo, Japan). Three scratches were created on the surface of each stone specimen with the surface property tester having a conical sapphire indenter with a 60° apex angle under vertical load at two different speeds. Scratches were created with scratching loads of 1N, 2N, and 3N, and scratching

Fig. 2 Three scratches produced on the surface of stone specimen and positions for measuring depth of scratches.

speeds of 50 and 100 mm/min. Using the profilometer, the depth of scratches was measured by a stylus tip with measuring force of 0.7mN at a tracing speed of 0.3 mm/sec. As shown in Fig. 2, three measurements were obtained for each scratch at three different positions. The mean of nine measurements for each specimen was adopted as the representative value. The scratch depth value was defined as the scratch hardness. Six specimens were prepared for each condition. Scratch depth data were subjected to analysis of variance with factors of scratch load, scratch speed, and storage period, and Tukey's multiple tests for statistical comparison of the measuring conditions ($\alpha = 0.01, 0.05$). In addition, the data were analyzed for differences between material types (AFIII+NP and EMF+NF) by *t*-test ($\alpha = 0.01, 0.05$).

Scanning electron microscope (SEM) observations

Surface of stone specimens was observed by scanning electron microscope (JSM-5300, JEOL, Tokyo, Japan) at magnifications of $\times 350$ and $\times 1,000$.

RESULTS

Scratch hardness (scratch depths) of the stone specimens are shown in Figs. 3 and 4. An increase in scratch depth indicates a decrease in scratch hardness. The results of ANOVA and Tukey's multiple tests for comparison of measuring conditions are summarized in Tables 2 to 4. The results of *t*-test showed significant differences between the scratch hardness of AFIII+NP and EMF+NF specimens under all measuring conditions. On this note, the results are not tabulated.

Scratch load

Scratch load significantly affected scratch hardness of AFIII+NP and EMF+NF specimens. The scratch depth of both specimens significantly increased with increase in load.

Scratch speed

Scratch speed did not affect scratch hardness significantly. However, an interaction between scratch load and speed was found for the AFIII+NP specimens. The results of Tukey's multiple tests for hardness of the AFIII+NP specimens indicated a significant difference between scratch speeds of 50 mm/min and

100 mm/min under a load of 3N for 24-hour storage and under a load of 2N for 1-week storage.

Storage period

Storage period significantly affected scratch hardness. The scratch depth of EMF+NF specimens stored for 1 week tended to be smaller than those stored for 24 hours at the scratch speed of 100 mm/min. Results of Tukey's multiple tests showed that significant differences between storage periods of 24 hours and 1 week occurred in EMF+NF specimens with scratch loads of 1N, 2N, and 3N at a speed of 100 mm/min.

Fig. 3 Scratch hardness of AFIII+NP specimens.

Fig. 4 Scratch hardness of EMF+NF specimens.

Table 2 Results of ANOVA for AFIII+NP and EMF+NF specimens

	Load	Speed	Storage period	L×S	L×P	S×P	L×S×P
AFIII+NP	**	—	*	**	—	—	—
EMF+NF	**	—	**	—	—	—	—

L×S: Interaction of load and speed

L×P: Interaction of load and storage period

S×P: Interaction of speed and storage period

L×S×P: Interaction of load, speed, and storage period

** : $\alpha = 0.01$; * : $\alpha = 0.05$; — : not significant

Table 3 Results of Tukey's multiple tests for comparison of measuring conditions for AFIII+NP specimens

			24-hour storage						1-week storage					
			50 mm/min			100 mm/min			50 mm/min			100 mm/min		
			1N	2N	3N	1N	2N	3N	1N	2N	3N	1N	2N	3N
24-hour storage	50 mm/min	1N		**	**	—			—					
		2N			**		—			—				
		3N						**			—			
	100 mm/min	1N				**	**				—			
		2N					**					—		
		3N												—
1-week storage	50 mm/min	1N							**	**	—			
		2N								**	*			
		3N												—
	100 mm/min	1N										**	**	
		2N											**	**
		3N												

** : $\alpha = 0.01$; * : $\alpha = 0.05$; — : not significant

Table 4 Results of Tukey's multiple tests for comparison of measuring conditions for EMF+NF specimens

			24-hour storage						1-week storage					
			50 mm/min			100 mm/min			50 mm/min			100 mm/min		
			1N	2N	3N	1N	2N	3N	1N	2N	3N	1N	2N	3N
24-hour storage	50 mm/min	1N		**	**	—			—					
		2N			**		—			—				
		3N						—			—			
	100 mm/min	1N					**	**				*		
		2N						**					*	
		3N												*
1-week storage	50 mm/min	1N							**	**	—			
		2N								**		—		
		3N												—
	100 mm/min	1N											**	**
		2N												**
		3N												**

** : $\alpha = 0.01$; * : $\alpha = 0.05$; — : not significant

Fig. 5 SEM images of the surfaces and scratches of AFIII+NP (a,b) and EMF+NF (c,d) specimens. Conditions: 1N load, 100 mm/min speed, 1-week storage.

Material type

The scratch depth of AFIII+NP specimen was significantly smaller than that of EMF+NF specimen under all conditions.

SEM observations

SEM images of the stone specimen surface and the scratch under conditions of 1N scratch load, 100 mm/

min scratch speed, and 1-week storage are shown in Fig. 5. SEM images of the stone specimen surface revealed only small differences in crystal structure between AFIII+NP and EMF+NF specimens. There were no differences in the SEM images of the scratch produced under varied conditions of scratch load, scratch speed, and storage period.

DISCUSSION

AFIII+NP and EMF+NF specimens stored for 1 week showed lower Knoop hardness values as compared with those of stones set on acrylic plate¹²⁾. Moreover, the indentation was not produced clearly. It is therefore necessary to give some treatment on the surface of stone models obtained from disinfected impressions in order to measure their Knoop hardness values. We conducted the hardness test for stone models by measuring scratch hardness with a clear indentation. The specimens were subjected to varying conditions of scratch load, scratch speed, and storage period. Their effects on the scratch hardness of stone models were evaluated to further investigation of the effect of disinfection of impressions on surface hardness of stone models.

Scratch load

Under all conditions, scratch depth of AFIII+NP and EMF+NF specimens increased significantly with increase in scratch load. Values for scratch depth at 1N, 2N, 3N loads produced ratios of 1:1.5-1.7:1.9-2.4. Contact area between the indenter and specimen surface increased with the square of scratch depth when a conical indenter with a 60° apex angle was used to form a scratch. Values for contact area at 1N, 2N, 3N loads produced ratios of 1:2.3-2.9:3.6-5.8. For Knoop hardness, the projected area is divided into the load to give the Knoop hardness number. Scratch hardness calculated using this formula yielded unconstant values. Therefore, it is considered necessary to obtain clear information on how scratch hardness value is derived. Although the test specimens in the present study had sufficient hardness because they were obtained from impressions without treatment of reducing the surface hardness of stone models, scratch depth could be analyzed statistically even at the lightest load (1N). In the case of 3N loads, the tip of the indenter became worn-out after prolonged testing.

Scratch speed

Scratch speed did not affect significantly the scratch depth of either the AFIII+NP or EMF+NF specimens. However, an interaction between load and speed was found for the AFIII+NP specimen. In the AFIII+NP specimen stored for 24 hours, scratch depth at a speed of 100 mm/min with 3N load was significantly smaller than that at 50 mm/min. In the AFIII+NP specimen stored for 1 week, scratch depth at a speed of 100 mm/min with 2N load was significantly larger than that at 50 mm/min. Li¹⁵⁾ reported that scratch depth decreased with an increase in scratch speed during hardness testing of soda-lime glass. In the present study, little difference existed in scratch depth between speeds of 50 mm/min and 100 mm/min, and the scratch depth at

100 mm/min was not always smaller than that found at 50 mm/min. This was probably because the set stone was different from soda-lime glass in that it was porous. It is evident that more experiments that involve different scratch loads and speeds are required.

Storage period

The compressive strength of set plaster increases with drying¹⁶⁾. A similar change in surface hardness has been reported to occur during the drying process¹⁶⁾. For the EMF+NF specimen, the scratch depth after 1-week storage exhibited a tendency to be smaller than that found after 24-hour storage at a speed of 100 mm/min. Indeed, the EMF+NF specimen showed a significant difference between 24-hour storage and 1-week storage at the scratch speed of 100 mm/min. This could be due to the slow drying process of NF as compared to NP.

Material type

Material specifications state that the compressive strength one hour after setting is smaller for NP than for NF (NP: 49.0 MPa; NF: 63.7 MPa). However, the scratch depth of AFIII+NP specimen was significantly smaller than that of EMF+NF specimen under all measuring conditions. The scratch hardness of stone models was reported to vary with the type of impression material¹⁴⁾. Scratch hardness of stone models is considered to be influenced by the combination of gypsum and impression material, suggesting that surface hardness and compressive strength are not closely related.

In this study we demonstrated that the scratch depth of stone models increased with increasing scratch load. And for Type 4 stone model obtained from vinyl silicone rubber impression, scratch depth after 1-week storage was smaller than that after 24-hour storage at a speed of 100 mm/min. However, the effect of scratch speed on scratch depth did not follow a specific trend.

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