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CHEMICAL DURABILITY IMPROVEMENT AND STATIC FATIGUE OF GLASSES.(U)
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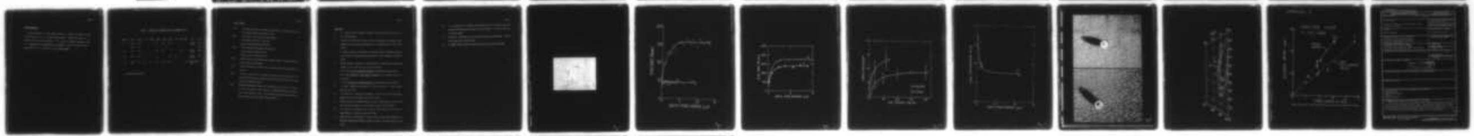

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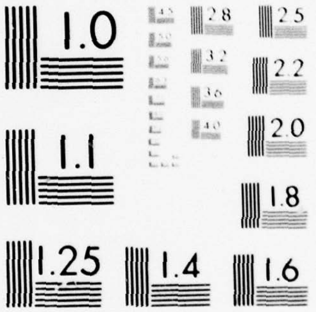
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For the Period Ending: February 28, 1979

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6 CHEMICAL DURABILITY IMPROVEMENT AND STATIC FATIGUE OF GLASSES.

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I. SUMMARY

The Surface layer of heat-treated borosilicate glasses exhibited lower HF etching rate compared with the bulk. The chemical analysis of the same glass indicated an excess Al_2O_3 concentration in the surface layer. The low HF etching rate was attributed to the lowering of the immiscibility dome by the excess Al_2O_3 and the consequent shift of the composition.

A prolonged heat-treatment of glasses produced a surface stress layer. This surface stress was found to change its sign when the glass was heat-treated in wet atmosphere. The surface stress was attributed to the different water content in the surface layer from that in the bulk.

Chemical durability, especially the etching rate of a glass in hot NaOH solution was found to be reduced by coating the glass with Zr alcoxide. The Zr compound appears to deposit on the etched surface continuously protecting the glass.

Direct confirmation of the stress corrosion, i.e., the stress-accelerated reaction of glass with aqueous solution was attempted. Preliminary investigation showed that reactions are accelerated by tensile stress and retarded by compressive stress.

II. RESEARCH AND RESULTS

1. Surface Structure of Glasses

The Surface layer of some commercial glasses was found to have different properties from those of the bulk. The origins of these phenomena were investigated.

(a) HF etch rate of phase separated glasses.

The Surface layer of heat-treated borosilicate glasses showed a higher chemical (HF) durability compared with the bulk. This surface layer was observed only for as-received glass rods and only for glass compositions which give the interconnected microstructure upon heat-treatment. The chemical analysis revealed the excess Al_2O_3 concentration in the surface layer. The Al_2O_3 is known to suppress the immiscibility tendency of borosilicate glasses. Thus the excess Al_2O_3 content in the surface layer is expected to reduce the composition range of the phase separation, reducing the alkali-borate concentration of the alkali-borate rich phase. Since the chemical durability is primarily controlled by the composition of the continuous alkali-borate phase, the higher chemical durability results.

(b) Surface stress

When a glass containing alkali was heat-treated for an extended period of time the surface layer showed a residual stress which cannot be eliminated by annealing. This stress layer was observed regardless of the presence of phase separation or of the prior etching of the original surface. The sign of the surface stress was observed to reverse itself when the glass was heat-treated in wet atmosphere. The origin of this surface stress was attributed to the decrease (or increase) of water content in the surface layer of the glass. Although the evaporation of Na from the glass surface can cause the similar stress, this effect was considered secondary because of the stress sign reversal due to wet atmosphere and the comparatively thicker stressed layer than the alkali deficiency layer.

These investigation on the glass surface will be published in July - August issue of the American Ceramic Society (1979). The manuscript is attached to this report as Appendix I.

2. Chemical Durability Improvement by Coating

ZrO_2 component in glass is known to improve the chemical durability of glasses substantially. Here coating of glass with Zr containing compounds was studied. Soda-lime slide glass was coated with Zr alcoxide by dipping it into the alcohol solution of Zr alcoxide. The etching rate of the slide glass in 10% NaOH at 80°C was determined by measuring the weight loss per unit surface area. With the Zr alcoxide coating the etching rate was observed to become lower by 20 ∿ 30%. This is shown in a figure as Appendix II. Similar result was obtained for fused silica also. Since the coating thickness is far less than the etched layer, Zr compound is probably seeking and depositing itself on the etched glass surface. To confirm this possibility, a small amount of Zr alcoxide was placed in NaOH solution and detching experiment was conducted and similar improvement in durability was observed. This result will be presented at the 81st Annual Meeting of the American Ceramic Society, to be held in May 1979, in Cincinnati, Ohio. At present, the exact mechanism of durability improvement by Zr alcoxide coating is being investigated.

3. Stress Corrosion

Even though the static fatigue of glass is believed to be caused by the stress accelerated reaction of glass with atmospheric water, very little experimental evidence exists on the stress accelerated reaction itself. Here, the direct study of the stress accelerated reaction of glass with various aqueous media is made. To this end, glass plate is subjected to the known amount of stress in a specially designed four point loading cell and is immersed in various aqueous solutions. So far preliminary results indicated that the ion-exchange reaction rate of water and alkali as well as the leaching reaction rate of a phase separated glass with HCl were stress dependent. With the aid of an ellipsometer, to be purchased, it is anticipated that the quantitative measurement of the reacted layer thickness will become possible and that the stress rate dependency of the reaction will be clarified.

III. PERSONNEL

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Associate Professor of Materials Engineering

Yoshio Oka
Postdoctoral Research Associate

Karl Ricker
Research Assistant

IV. PUBLICATION

"Relation of Surface Structure of Glass to HF Acid Attack and Stress State", M. Tomozawa and T. Takamori (to appear in J. Am. Ceram. Soc.).

V. ORAL PRESENTATION

"Chemical Durability Improvement by Coating", by K. Ricker, Y. Oka and M. Tomozawa (to be presented at the 81st Annual Meeting of the American Ceramic Society, May 1979, Cincinnati, Ohio).

APPENDIX I

To appear in
J. Am. Ceram. Soc.

RELATION OF SURFACE STRUCTURE OF GLASS TO HF ACID ATTACK AND STRESS STATE

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ABSTRACT

The surfaces of commercial glasses, mainly borosilicate glasses, were investigated by HF etching and surface stress measurement. The HF etch rate was slow at the surface for some phase-separated glasses; this was attributed to excess alumina in the surface layer produced during the glass manufacture. After heat treatment of the glass rods either with or without the as-received surfaces, compressive (tangential) stresses were observed in all the glasses studied. This is most likely due to the evaporation of water from the surface during the heat-treatment.

I. INTRODUCTION

The surface condition of glasses has a strong influence on the chemical and mechanical properties of the bulk samples. Hood and Nordberg[1] noted during the production of Vycor-type glasses that the leaching process of a phase-separated borosilicate glass is hindered by the surface layer; if the original surface layer is removed prior to heat treatment this effect is almost completely eliminated. Eguchi et al.[2] established for a glass [illegible] a ternary Na_2O-B_2O_3-SiO_2 composition that selective evaporation of the components, Na_2O and B_2O_3, takes place from the hydrated surface during heat treatment, leaving a silica layer of up to 1000Å thickness on the glass surface. A much thicker surface layer of 100 μm was reported by Utsumi et al.[3] on a phase-separated glass of similar composition on the basis of bending strength measurements. In the course of a study of thermal strains Takamori[4] observed the formation of a surface layer after the heat treatment for phase separation.

In the present work, two experimental methods, i.e. chemical etching rate and stress measurements, are employed to reveal surface characteristics different from the bulk properties of selected commercial glasses, particularly in the case of phase-separated borosilicate glasses. An attempt is made to clarify the separate roles of various components in the formation of such surface layers.

II. EXPERIMENTAL PROCEDURE

The compositions (mostly obtained from the published literature[5]) of the commercial glasses used in the present experiments are shown in Table I. Glasses I to III are borosilicate glasses which can be phase-separated by appropriate heat treatments, while Glass IV is a soda-lime glass which has no tendency toward phase separation. As-received glass rods of 4-6 mm in diameter were cut into an approximate length of 60mm and heat-treated in air at 600°C for various lengths of time to induce phase separation (where possible), and were furnace cooled (cooling rate ≈50°C/hr). For comparison, some samples were simply annealed by furnace-cooling from their annealing temperatures. The HF etch rate of the surface layer of the heat-treated glass specimens was determined as a function of the depth from the glass surface. Each specimen was exposed to the etching solution, 10% or 2% HF, for 30-60 sec. at 20 ± 1°C and was washed with alcohol. The etch rate was determined from the weight change per unit area and the density of the glasses.

After heat treatment the surface layer (to a depth of 50-100μm) showed compressive (tangential) stresses,[4] as illustrated in Fig. 1 for a cross section of Glass I rod. This surface stress on the rods of 6mm in diameter was measured as a function of heat treatment time by a polarimeter.* The chemical composition of the surface layers was determined for a selected glass specimen as a function of the depth from the surface. Specifically for Glass I, Si and K were analyzed with an electron microprobe analyzer, Na and Al with an atomic absorption spectrometer. For the latter, sample rods were etched off with 10% HF successively from the surface, and the Na and Al contents in each HF solution were determined as a function of the depth from the sample surface. The similar measurements were made for Na and Ca in Glass IV. Two stage replica electron microscopy[6] was used to look into the microstructures.

III. RESULTS

(1) Etch Rate Measurement

Typical results of chemical durability are shown in Figs. 2, 3, and 4 for Glass I, II and III, respectively. The HF etch rate of Glass I and II increases with the depth from the surface after the glasses were heat-treated for phase separation (Fig. 2, curve B, and Fig. 3). The specimens simply annealed show a constant etch rate (Fig. 2, curve A). Thus in Glasses I and II, surface layers 4-5 μm thick have a different chemical durability from the bulk. This

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*Model C36 comparator polarimeter, Polarizing Instrument Co., Inc., Irvington-on-Hudson, New York.

difference is revealed only after the specimens were heat-treated for phase separation. The thickness of the surface layer was found to vary slightly depending upon the specimen lot as well as on the specimen diameter. When the surface layer of the as-received glass rods was etched off prior to the heat treatment, this change of the HF etch rate with depth was almost entirely eliminated.

In contrast to Glasses I and II, in Glass III the etch rate is independent of depth, even after the heat treatment for phase separation. This is also true for Glass IV, which does not phase separate.

(2) Stress Measurement

The surface stress in all four glasses examined after the heat treatment for phase separation was found to increase with the duration of the heat treatment. As shown in curves A and B in Fig. 4 for Glass I, the magnitude of the surface stress varies from lot to lot. In contrast to the etch rate, the surface stress produced by the heat treatment was not altered by the elimination of the original surface layer (through HF etching) prior to the heat treatment. The thickness of the stressed surface was estimated to be ~ 100μm.

(3) Surface Composition and Structure

The concentrations of selected components (Si, K, Na) in the surface layer of the heat-treated Glass I as a function of the depth from the surface were found constant within experimental errors. On the other hand, the $A\ell$ concentration was found to be higher in the surface layer of Glass I, as shown in Fig. 5. The concentration of Na and Ca in Glass IV showed a slight decrease in the surface layer of $\approx 2\mu$m depth after heat-treatment at 550°C for 100 hrs., similarly to the reported data by Sieger[7].

The electron micrographs of fractured and etched surfaces of Glasses II and III shown in Fig. 6 (A) and (B) provide evidence about the structure of these glasses. The heat treatment for phase separation induces an interconnected structure in Glass II (Fig. 6 (A)), and a discrete structure in Glass III (Fig. 6 (B)).

IV. DISCUSSION

Only some of the phase-separated glasses exhibit a variation in etch rate as a function of depth from the surface. However, in all glasses an increase in heat treatment time results in a corresponding increase in surface strain. Since it is evident that the two phenomena do not share the same origin, they will be discussed separately.

(1) Etch Rate

A) Effect of Microstructure

Among various commercial borosilicate glasses, Glasses I and II showed a surface layer with an etch rate different from that of the inside, while Glass III did not. This etch rate characteristic correlates well with the microstructure of phase separated glasses. Simmons et al.[8] observed an interconnected microstructure for Glass I heat-treated at 600°C; Fig. 6 (A) shows that Glass II also displays an interconnected structure upon phase separation. With this type of microstructure, the HF etch rate of the bulk sample has been shown to vary with composition changes of the chemically less durable (lower SiO_2 content) phase.[9] On the other hand, Fig. 6(B) shows that Glass III produces a dispersed minor phase embedded in a silica-rich matrix. With this microstructure, the HF etch rate of the bulk sample has been shown[9] to remain essentially constant through the progress of phase separation.

B) Composition Shift

As mentioned above, an excess of Al_2O_3, apparently produced during glass manufacture, was observed in the surface layer. (The density change caused by this composition change is small, therefore the etch rate measurement in Figs. 2, and 3 is not affected.) The observed variations in the surface etch rate can be explained by this high concentration of Al_2O_3. It is known that the addition of small amounts of Al_2O_3 to alkali-borosilicate glass[10] system as well as alkali silicate systems[11] suppresses the immiscibility dome. The surface layer with higher Al_2O_3 content will therefore have smaller composition variations than the bulk when heat-treated at the same temperature. Hence, from the direction of the tie line in the phase diagram,[9,12] the composition of the chemically less durable phase in the surface layer will have a higher SiO_2 content than the bulk. This is illustrated schematically in Fig. 7. Since the HF etch rate of these phase separated glasses I and II is primarily controlled by the SiO_2 content of the chemically less durable phase,[9] after phase separation the surface layer will have a lower etch rate than the bulk. For Glass III, the shift of composition of the chemically less durable phase has no effect on the HF etch rates because of its microstructure with discrete second phase particles.

Alkali oxides and boron oxides are known to volatilize together[13]. Since the analytical results indicate no alkali deficiency in the surface of the borosilicate glass rods it is anticipated that there is also very little boron deficiency. Furthermore, the shift of the chemically less durable phase composition would be small even if the slight boron deficiency existed (cf. Fig. 7).

(2) Surface Stress

Surface stresses were produced by an extensive heat treatment regardless of whether or not the specimen is phase separated. Since the prior etching of the surface of glass rods did not cause any change in the surface stress, it is apparent that variations of composition in the surface layer produced during manufacturing is not the cause of the stress. The decrease in concentration of Na in the surface layer observed in heat-treated Glass IV may be involved in the surface stress formation. The alkali deficiency would reduce the thermal expansion coefficient of the surface layer producing the surface compressive (tangential) stresses which are observed. This possibility can not be rejected since a glass specimen with no alkali component showed practically no surface stress after the heat-treatment.

However, the major cause of the surface stress formation has to be looked for elsewhere, since no alkali deficiency was observed in Glass I after the heat-treatment and also the thickness of the surface strain layer was far greater ($\approx 100 \mu m$) than the thickness of Na deficiency layer ($\approx 2 \mu m$) observed in Glass IV after heat-treatment.

It is suggested that the cause of the surface strain is the difference in water content between the surface layer and the bulk. The specimen heat-treated in dry atmosphere is expected to have a low water content in the surface layer. The low water content in the surface layer of glass leads to the low thermal expansion[14] (or contraction) which, upon cooling, produces surface compressive (tangential) stresses.

To test this hypothesis, samples of Glasses I and IV were heat-treated in an atmosphere of water vapor in order to increase the water content of the surface layers. As expected, the resulting surface stresses were reversed in sign in comparison with the surface stresses observed in specimens heat-treated in air. Thus, the compressive stress in the surface of glasses after an extensive heat treatment in air can be attributed to a decrease of water content due to evaporation.

Acknowledgement

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TABLE I CHEMICAL COMPOSITION OF GLASSES (WT %)

GLASS	SiO_2	B_2O_3	CaO	MgO	Al_2O_3	Na_2O	K_2O	Li_2O	PbO	BaO	Code	Ref.
I	80.5	12.9			2.2	3.8	0.4				Corning 7740	5(a)
	80	13			2	4						5(b)
II	73.0	16.5			2.0	4.5			4.0		Corning 7720	5(b)
III	70.0	28.0			1.1		0.5	1.2			Corning 7070	5(a)
IV	69.0	1.8	4.2	4.1	2.4	16.1	0.5			1.9	Owens-Illinois R-6	•

- • Chemical analysis at IBM.

Figure Captions

Fig. 1 An example of surface stress observed by polariscope. Cross-section of Glass I, heat-treated at 600°C for 100 hours, and rate.

Fig. 2 10% HF etch rate of the surface layer of Glass I
(A) annealed at 550°C for 1 hour, and
(B) heat-treated at 600°C for 24 hours.

Fig. 3 2% HF etch rate of the surface layer of Glass II
(A) heat-treated at 600°C for 2 hrs.
(B) heat-treated at 600°C for 11 hrs.

Fig. 4 Surface stress vs. heat-treatment time at 600°C for Glass I. A and B refer to two different lots of the same glass.

Fig. 5 Al_2O_3 concentration as a function of the depth from the surface for Glass I, as received.

Fig. 6 Two-stage replica electron micrograph of fractured and etched surface of (A) Glass II and (B) Glass III, both heat-treated at 600°C for 100 hours. The latex sphere has a diameter of 0.5μm.

Fig. 7 Metastable immiscibility boundary of SiO_2-B_2O_3-Na_2O system (wt%, after Ref. 9). Dotted line indicates a likely position of the immiscibility boundary at 600°C with a small addition of Al_2O_3. The chemically less durable composition will move from point A to B with Al_2O_3 addition at 600°C.

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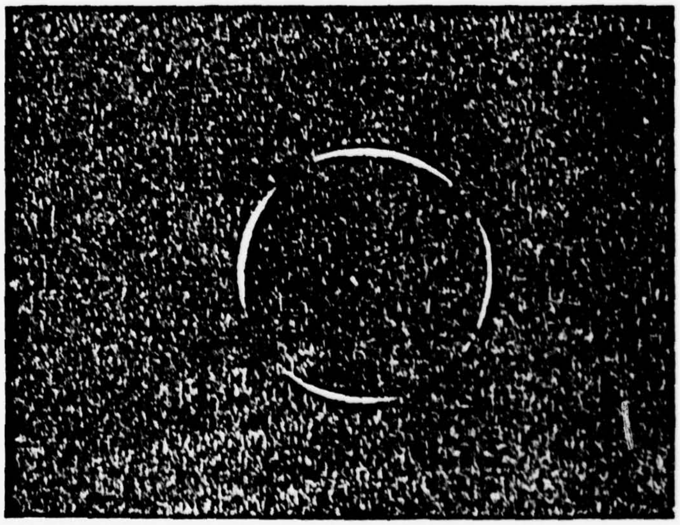

Fig 1

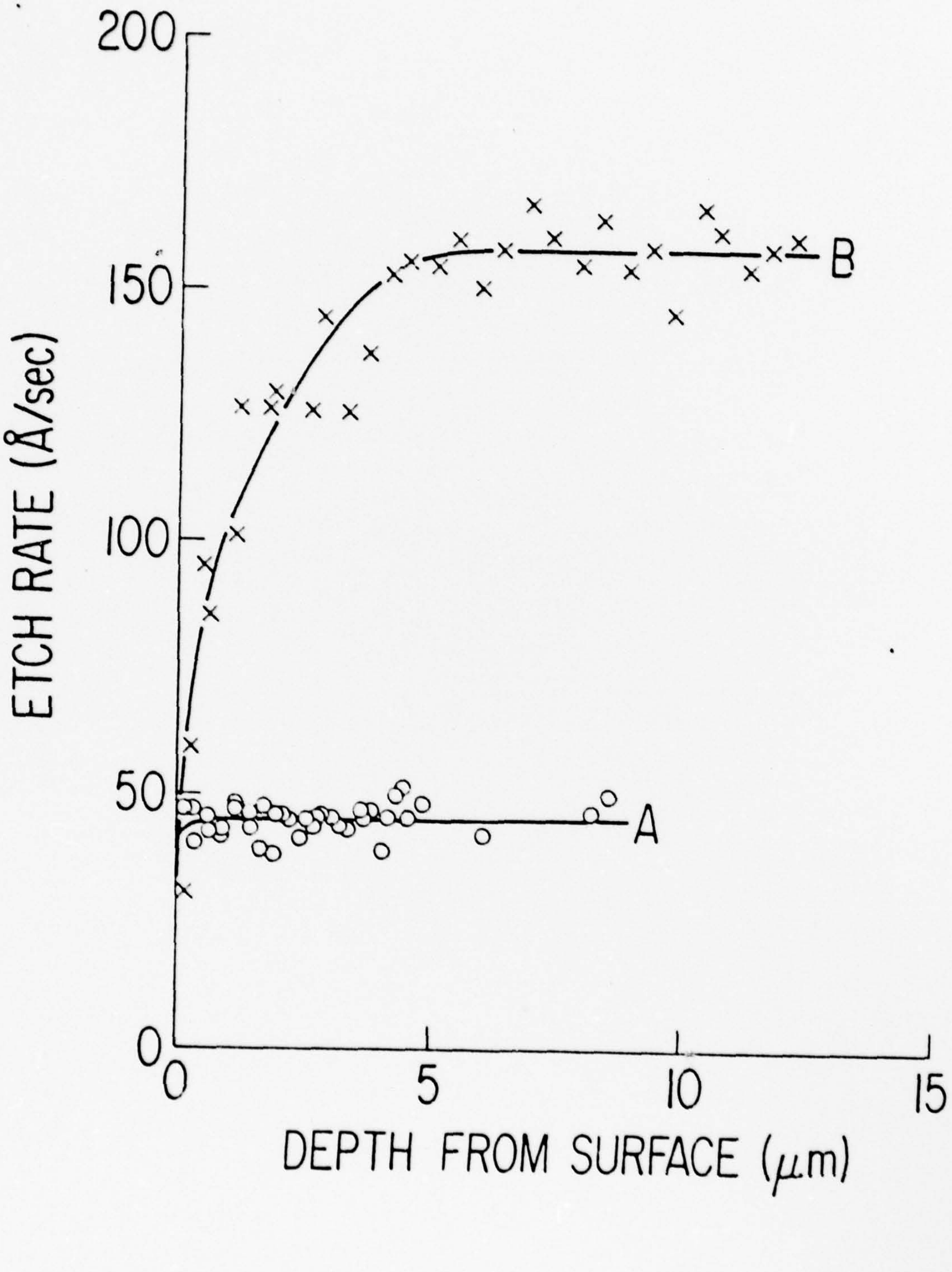

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Fig 2

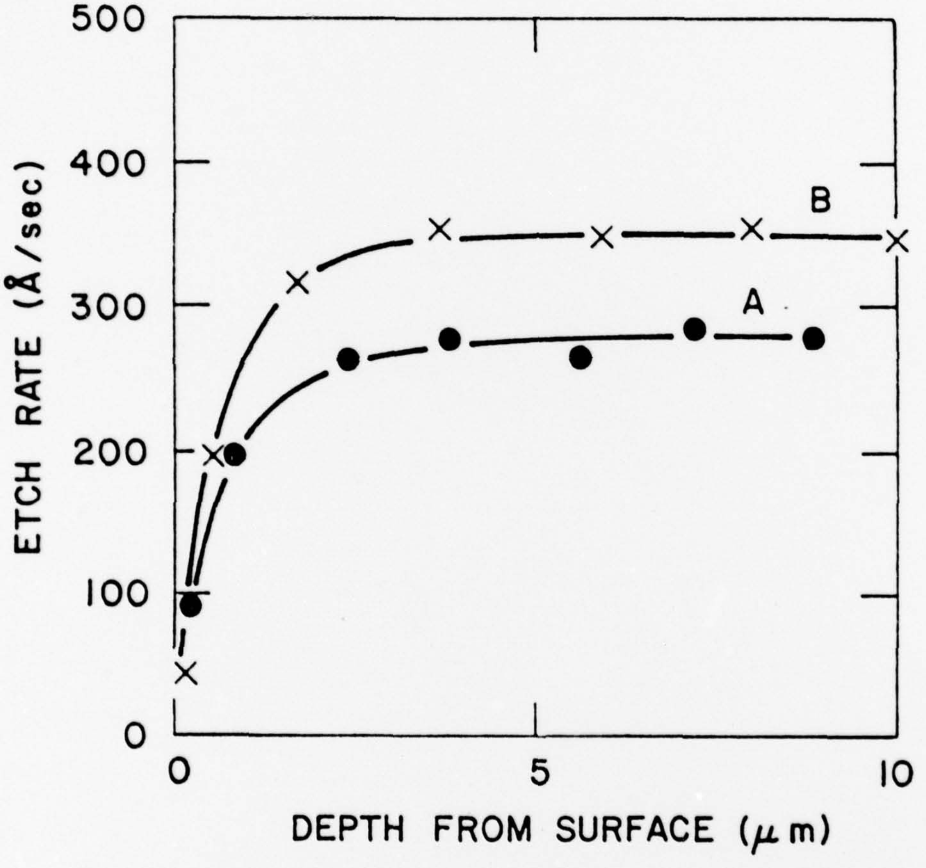

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Fig 3

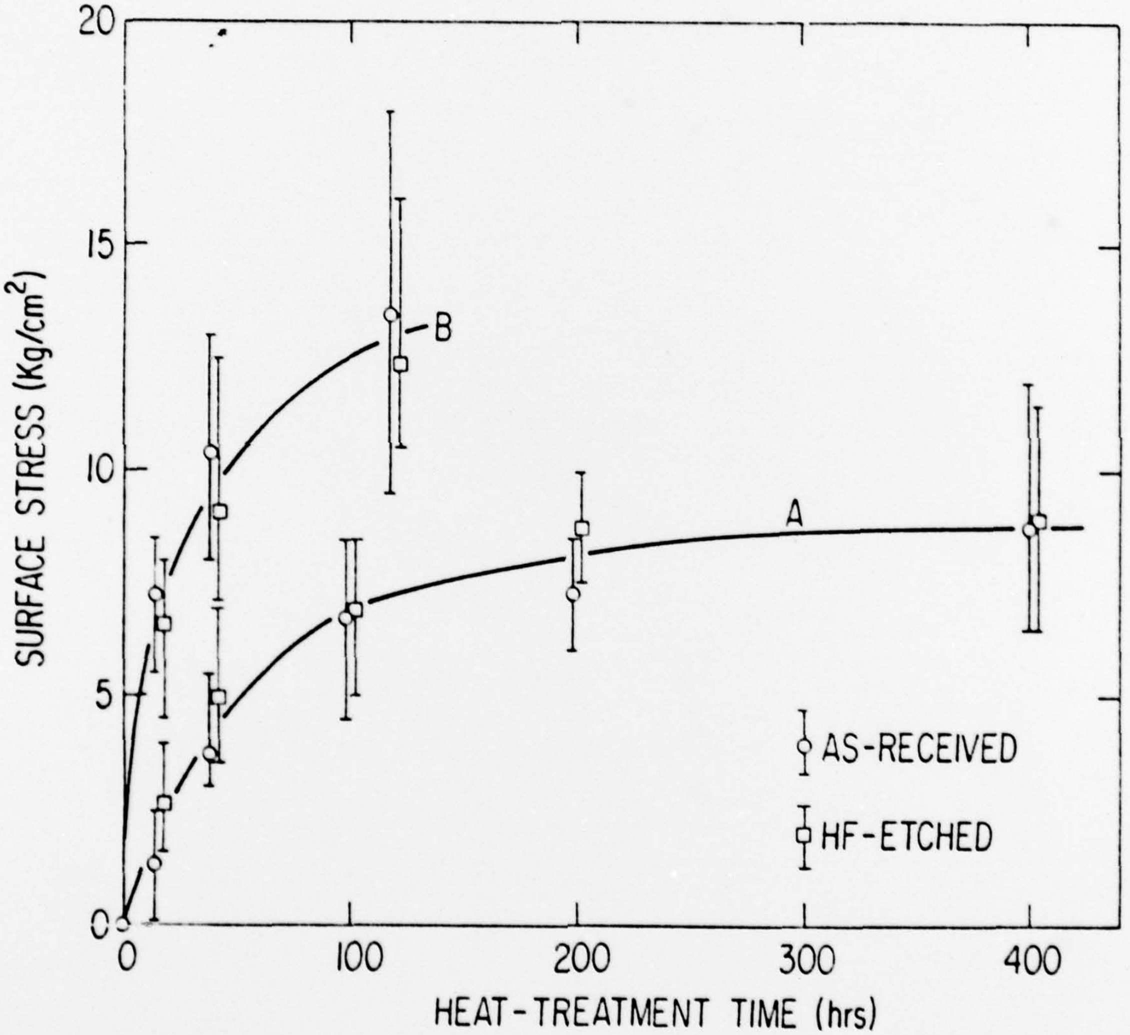
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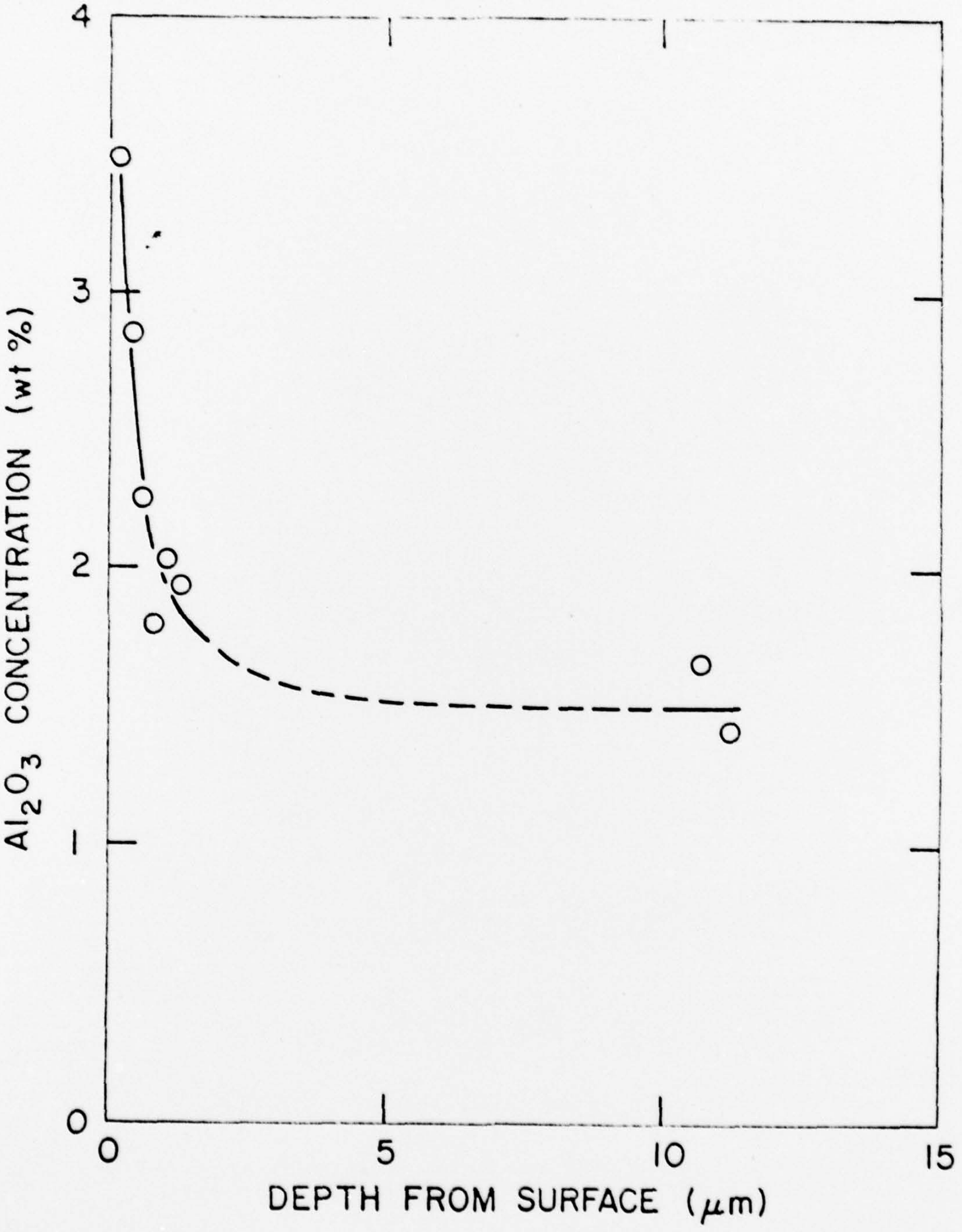
Fig. 4



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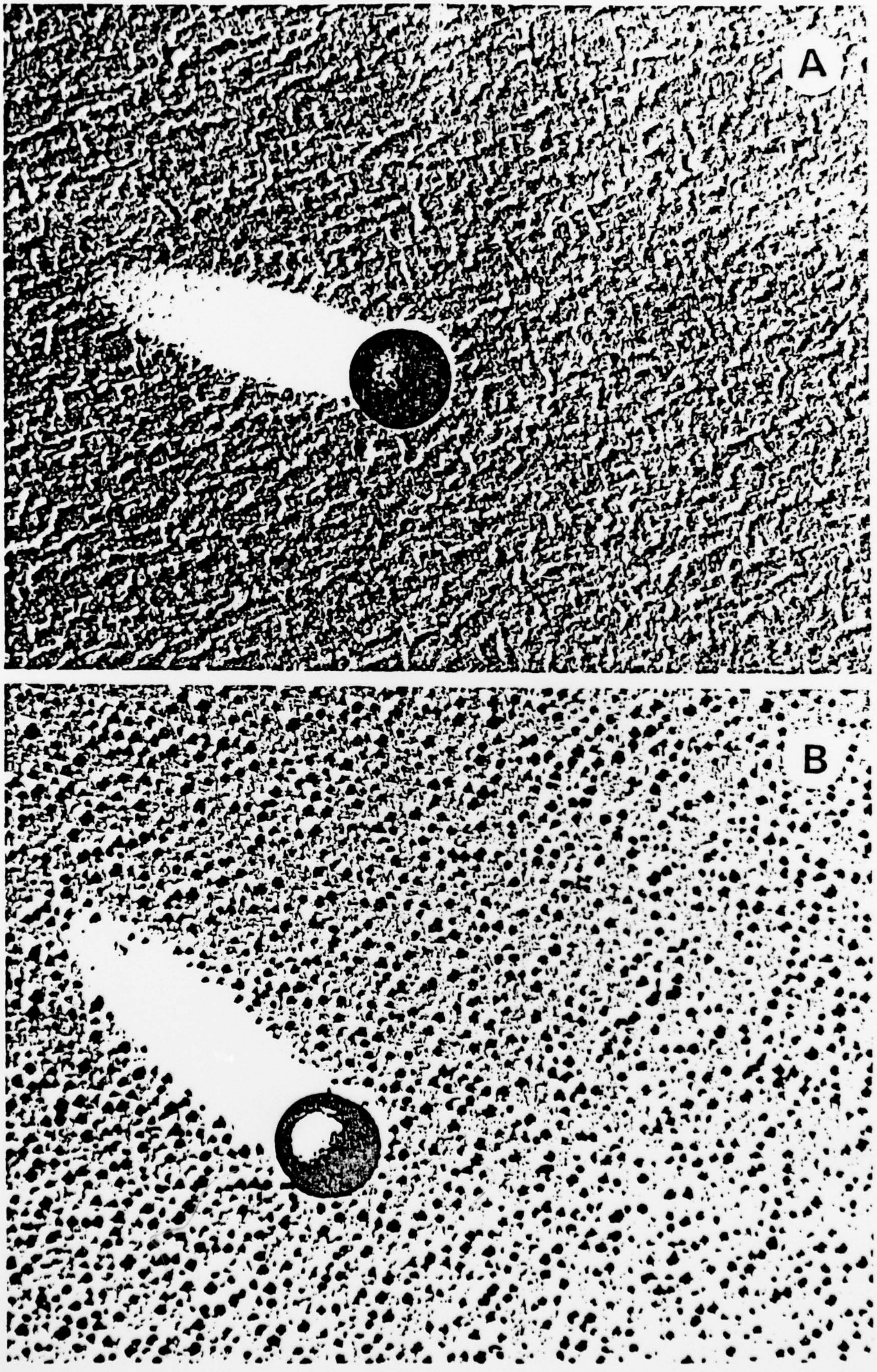

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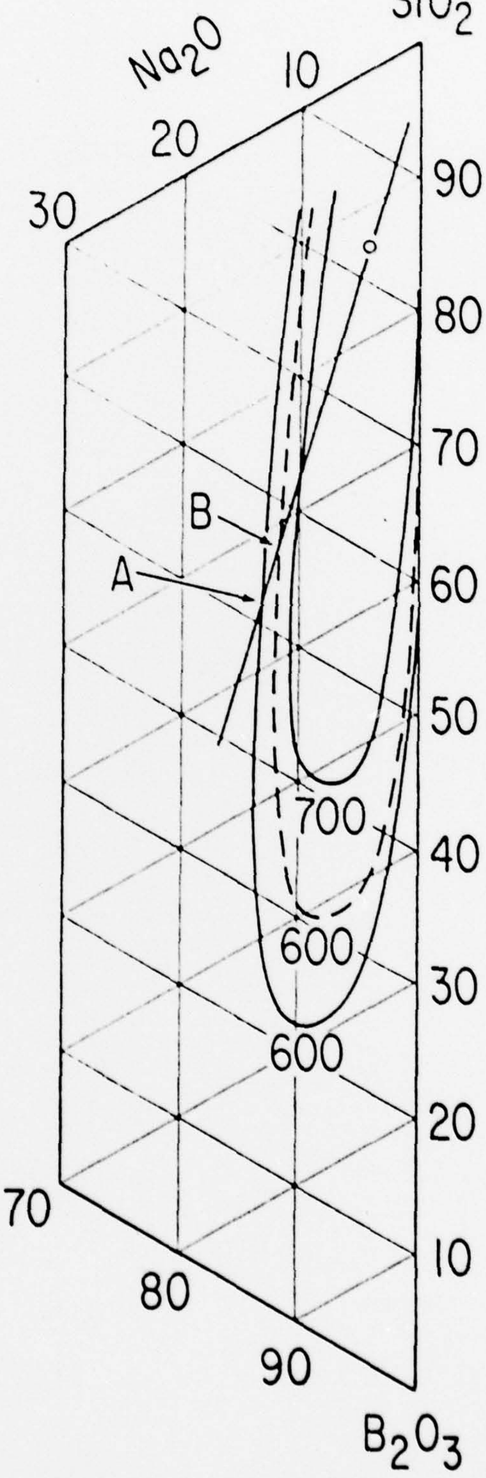
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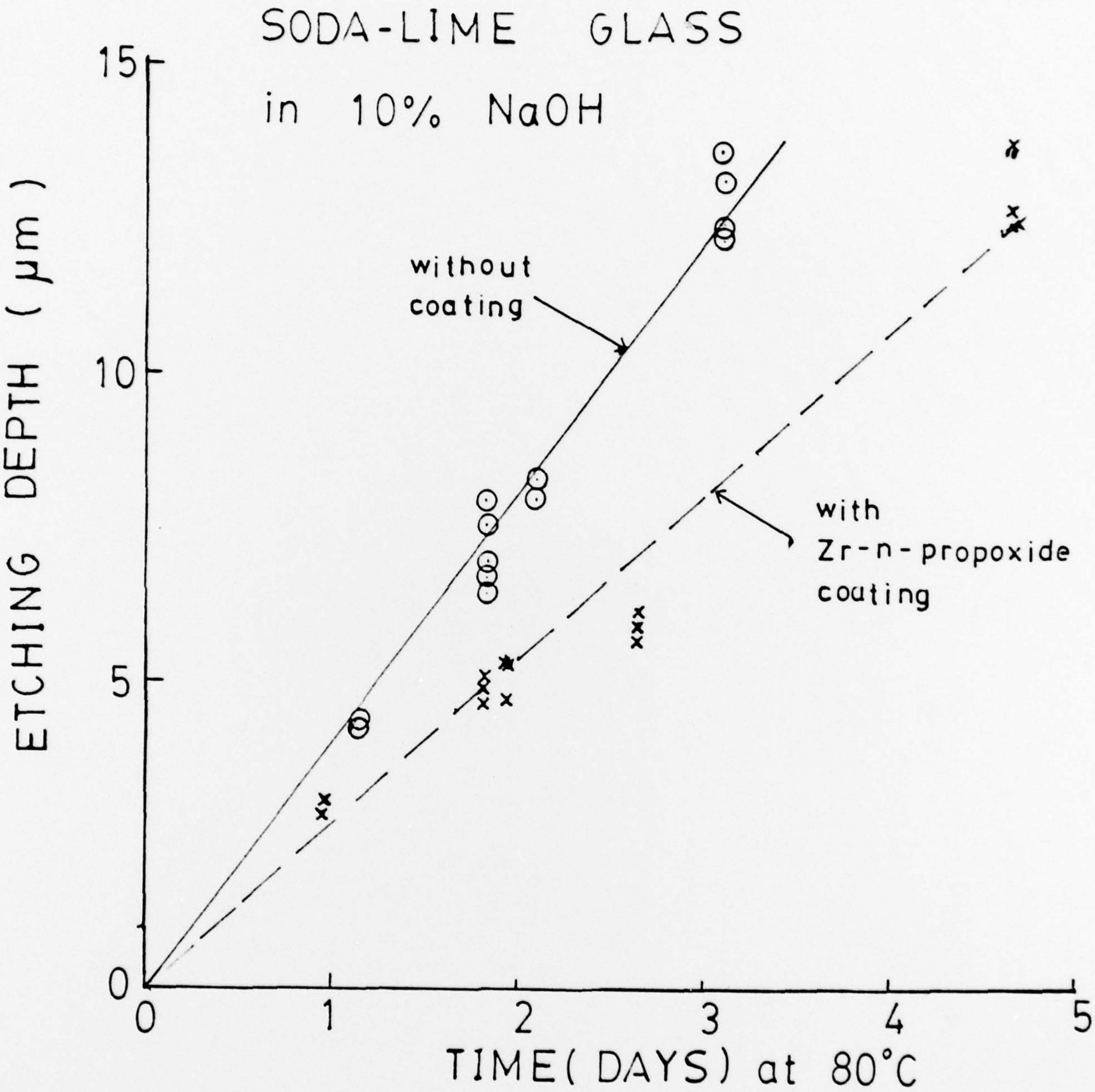
Fig 7

APPENDIX II



NaOH etching rate of soda lime glass with and without Zr-alcoxide coating.

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20. Abstract (continued)

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