JOURNAL OF MATERIALS SCIENCE 17 (1982) 1493–1500

Crack evolution in Vickers indentation for soda-lime–silica glass

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The sequence of radial and median crack formation during Vickers indentations on soda-lime–silica glass with different surface treatments has been investigated. For chemically-toughened soda-lime–silica glass no radial/median cracks were observed around indentations at loads high enough to induce cracks in annealed glasses, even though the thickness of the compressive stress layer was much smaller than the size of the deformed zone. This indicates that embryonic crack initiations at the surface are essential for the development of both median and radial cracks. The crack initiation was discussed from the point of view of the Perrott stress field model and SEM studies.

1. Introduction

Generally, fracture strength of glass is dominated by the surface flaws produced by contact with some other hard materials either during fabrication or in service. Two main simplified systems of "blunt indenter" and "sharp indenter" concerned with such crack formations have recently been studied [1–5]. In the blunt indenter experiments, for example, a Hertzian crack is typically introduced as a result of elastic fracture, the formation mechanism of which have been reviewed in detail for brittle materials by Lawn and Wilshaw [2]. For the sharp indenter system, however, rather complicated crack formations have been reported owing to elastic–plastic fracture beneath the indenter [1]. The main cracks induced in the sharp indenter system are the radial, median, and lateral cracks. The radial and the median cracks, particularly, are practically important, since they cause strength degradation of glass components in service [5].

The radial cracks in glass are produced at relatively low indentation load, and are slightly different from the Palmqvist cracks [4] which have been reported to be the only cracks observed in reasonably tough materials. Although the Palmqvist crack cannot extend beneath the deformed zone, the radial crack in soda-lime–silica glass can extend deeper than the deformed zone [6]. Hagan and Swain [7] have stated that the radial crack initiates from the glass surface and develops during the unloading half-cycle. While in the higher indentation load region the median crack grows from the sub-surface of the glass during the loading, and then fully develops into a semi-circular flaw during or after the unloading [1, 8].

However, in soda-lime–silica glass an indistinguishable load region between the radial and the median cracks exists where the indentation-induced cracks show continuous changes of the arrest position marks with the magnitude of the applied load [9]. In this study, therefore, we investigated the crack formation mechanisms in Vickers indentation for the radial and the median cracks by changing the applied load and the surface conditions of the glass specimen.

2. Experimental procedure

2.1. Glass samples

A commercial float glass* with soda-lime–silica composition was used in this work. The test specimens were cut from a sheet into laths, of dimensions 10 mm × 80 mm × 3 mm, for the convenience of bend tests.

In order to investigate the effects of the glass surface on crack formation in Vickers indentation, various samples listed in Table I were prepared: as-received, acid-etched abraded and chemically-

*FL-3 (3 mm thick), from Asahi Glass Co. Ltd, Tokyo.

0022–2461/82/051493–08$03.46/0

TABLE I Glass samples with various surfaces

Sample	Remarks
As-received	Top side of float glass
Acid-etched	30 μm surface layer removed
Abraded	Ground with Number 2000 SiC powder
Chemically-toughened	Immersed in KNO_3 at 450° C for 1 h

toughened specimens. As to the as-received specimens, the top side of float glass, distinguished by identifying the bottom side (tin-rich) with a u.v. light, was subjected to the indentation tests. The acid-etched specimens provide almost flawless surfaces with approximately 30 μm of the surface layer removed with a 5 wt% HF and H_2SO_4 mixed solution. The abraded samples were prepared by uniformly grinding the float glass with Number 2000 SiC powder, providing the surfaces with many minute flaws. In the case of the chemically-toughened glass, the acid-etched glasses were immersed in molten potassium nitrate at 450° C for 1 h to form extremely thin and high surface compressive stress layers (5 μm deep and more than 1000 MPa compressive stress at the surface). The depth and the surface compression of the layer were determined with a polarizing microscope equipped with a Berek compensator [10].

2.2. Indentation experiments

Indentation tests were carried out using a microhardness tester* with a Vickers diamond indenter in air at room temperature (18 to 23° C, 40 to 50% r.h.). The indenter load was varied from 2.45 to 24.5 N (250 to 2500 g), and applied over a 15 sec interval. The Vickers impression and the indentation-induced cracks were observed with a scanning electron microscope (SEM) and a differential-interference optical microscope (DIOM).

2.3. Bend tests

To confirm the existence of the indentation-induced cracks, 4-point bend tests (supporting span: 60 mm; loading span: 20 mm) for the indented specimens were made with an Instron-type testing machine†. The specimens were broken with one diagonal of the Vickers impression perpendicular to the tensile stress. The bend tests were carried out in liquid nitrogen to avoid moisture-assisted slow crack growth from taking place during the testing. The fracture surfaces of the specimens were also examined with SEM and DIOM.

3. Results and discussion

3.1. Crack morphology

Fig. 1 shows the fracture surfaces exhibiting the cracks produced in Vickers indentation in a loading range of 2.45 to 19.6 N for the as-received specimen. Various arrest position marks clearly appear on the fracture surfaces. Since the formation of those marks have been reported to be due to a slight change in the direction of the principal stress perpendicular to the median plane when crack evolutions occur discontinuously [11], the crack development processes can be elucidated from them.

At low indentation loads, 2.45 to 9.80 N, the first arrest position mark (abbreviated as 1st AM in Fig. 1) corresponding to the outer boundary of the indentation-induced crack shows complete semi-circular form. As the indentation load is increased, from 14.7 to 19.6 N, the shape of the first arrest position mark gradually changes from semi-circular into circular. At a load of 19.6 N the first arrest position mark develops into a typical circular shape, as shown by Lawn and Swain [1].

Fig. 2 schematically illustrates the distribution of the principle stress normal to the median plane when a Vickers indenter is pressed on the glass surface [8]. The tensile stress field extends downward from beneath the indenter, and the compressive stress field is produced near the surface during the loading half-cycle (see Fig. 2a). The crack development during the loading half-cycle is, therefore, limited to the circular shape because the extension of the crack at the surface is disturbed by the compressive stress field. While at the unloading stage, the compressive stress gradually disappears and the tensile stress covers around the indented area owing to the irreversively-compressed zone beneath the indenter (see Fig. 2b), so that the crack developed after the unloading takes the semi-circular form [8].

From the correspondence of the first arrest position marks to the stress field, the indentation-induced cracks can be classified into three types, as illustrated in Fig. 3. Type I is the semi-circular shape which is produced at low indentation loads and develops after the unloading. Type II is formed during the unloading in the middle indentation load region, showing a transient shape from

*Model MVK-E, from Akashi Manufacturing Co. Ltd, Tokyo.
†Model UTM-500I, from Toyo-Boldwin Co. Ltd, Tokyo.

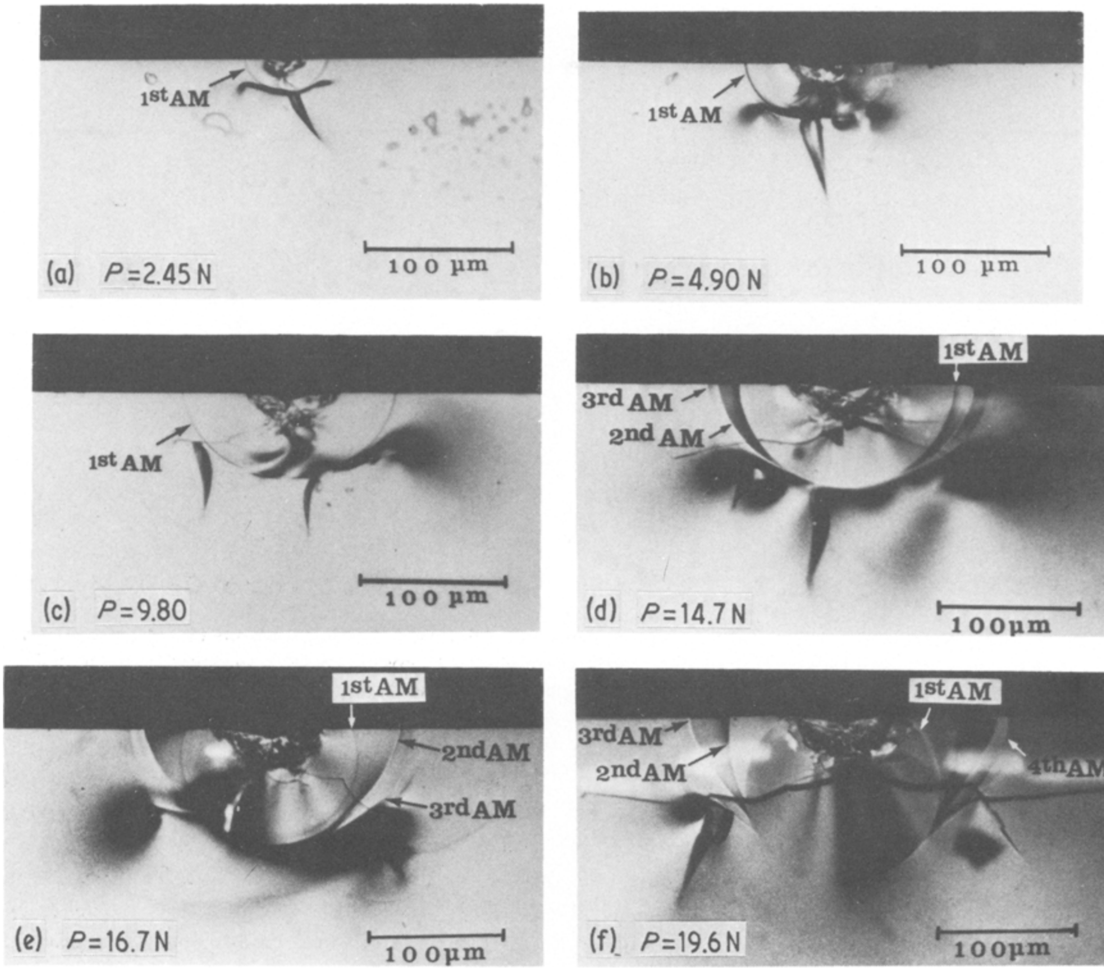


Figure 1 (a to f) Sub-surface views of soda-lime–silica glass indented with a Vickers diamond pyramid at various loads, P (AM indicates arrest position mark).

semi-circular to circular. Type III is observed only at higher indentation loads and shows an almost circular form which indicates that it develops during the loading half-cycle. Thus, the first arrest position marks reflect the stress field when cracks evolve.

According to Lawn and Swain [1], the median crack shows circular arrest position marks. In this work the typical median crack was observed only at 19.6 N, since the crack surface showed a circular first arrest mark. Crack systems for 2.45 to 9.80 N, illustrated in Fig. 1, can be considered to be radial cracks, because any circular arrest position marks do not appear on the crack surface and ledges can be seen which have been reported by Lankford and Davidson [12] to be a typical morphological feature for the radial crack system. However, crack systems for 14.7 N and 16.7 N are not definite at present. It can only be said that their first arrest mark takes a transient form from semi-circular to circular.

3.2. Effects of surface condition on crack initiation

Since the transient shape of the Type II arrest position mark indicates that the first arrest position mark varies continuously with the magnitude of the applied load, the crack development until the boundary indicated by the first arrest position mark may be interpreted to occur in same fashion regardless of the magnitude of the applied force. Further, considering that the initiation of the crack in the lower load region starts from the glass surface [6], it seems that the glass surface in contact with a Vickers indenter plays an important part in the crack evolution, even if at high indentation loads.

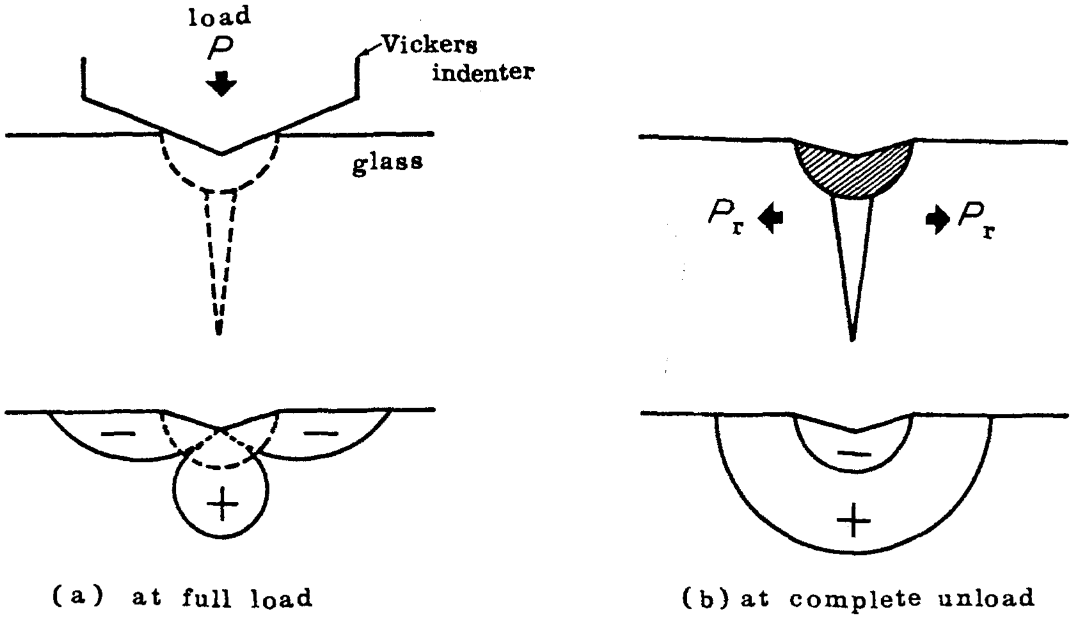


Figure 2 Schematic view of the distribution of the principal stress normal to the median plane during (a) loading and (b) after loading. The negative and positive signs mean compressive and tensile stresses, respectively [8]. The residual centre-opening force, P_r, in (b) results from the radially-expanded deformed zone (hatched part).

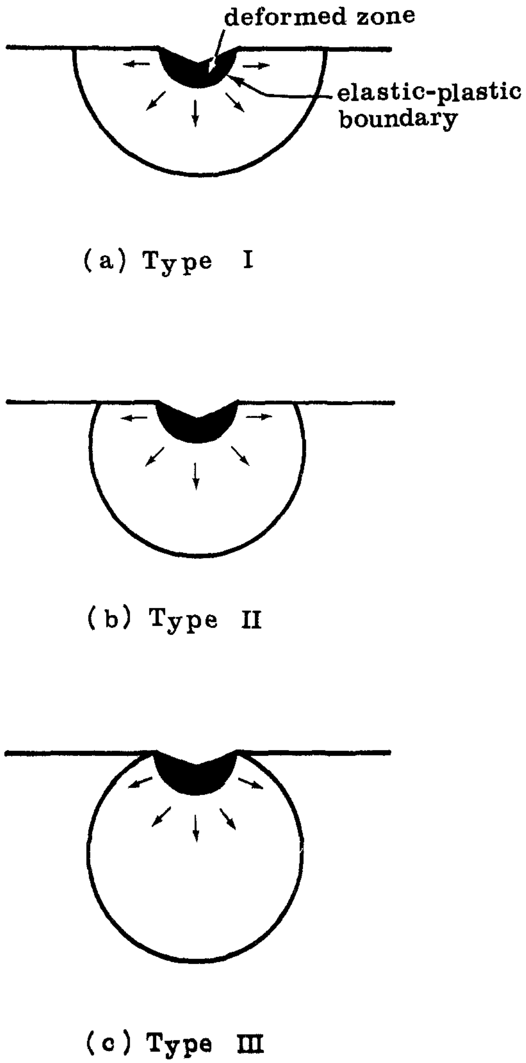
On the basis of these considerations, indentation tests were carried out on various specimens with different surface conditions, as listed in Table I. Each specimen was indented at a load of 9.80 N in air at room temperature and, subsequently, the Vickers impression at the surface was observed with DIOM. As summarized in Table II, radial cracks were found immediately after the indentation in the three groups of the as-received, the acid-etched and the abraded specimens. However, the chemically-toughened specimen with the very thin but high surface compressive stress layer showed the impressions without any cracks except only for one case with radial cracks just after the indentation, although the several impressions evolved the radial cracks after ageing in air for 24 h. These results mean that the crack evolution in Vickers indentation is strongly affected by the surface compressive stress, and is independent of the pre-existing surface flaw condition. Marshall and Lawn [13], and Swain *et al.* [14] have also reported effects of the surface compressive stress of thermally-toughened glasses on extension of the surface crack from the corners of the remnant impression. However, chemically-toughened glass has not been well studied, mainly because of its high compressive stress and severe stress gradient. In order to confirm the formation of the cracks for the chemically-toughened glasses the indented specimens were broken with 4-point bending in liquid nitrogen. The measured strength for the chemically-toughened specimens without the radial cracks was 703 ± 37 MPa which is considerably higher than those obtained both from the chemically-toughened specimens with the radial cracks and from the as-received specimens, as given in Table III, indicating that their high strength clearly reflects the non-existence of cracks of substantial size produced in the indentation.

Fig. 4 shows the fracture surfaces of the chemically-toughened glass both with and without the radial cracks. Fig. 4a, showing the glass with the existence of cracks, exhibits the semi-circular arrest position mark belonging to the Type I crack, and some distortion can be seen

TABLE II Radial crack evolution by Vickers indentation at 9.80 N in air at room temperature (20° C, 45% r.h.)

Sample	Percentage of radial crack evolution (%)*	
	Immediately after loading	Ageing for 24 h in air
As-received	100	–
Acid-etched	100	–
Abraded	100	–
Chemically-toughened	3.3	23.3

*Percentage for 30 indentations.



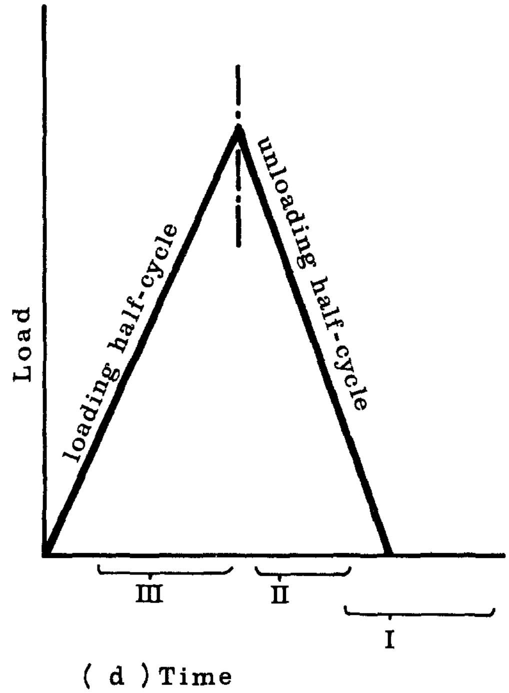


Figure 3 The three types of the first arrest position marks are shown in (a), (b), and (c) in Vickers indentation, and their formation periods with respect to the loading process are shown in (d).

in the near-surface region due to the surface compressive stress layer. The fracture surface without the cracks, shown in Fig. 4b, exhibits a small fracture mirror owing to the high fracture strength [15]. The irregular shape of the fracture mirror indicates that both corners of the Vickers impression at the surface are fracture origins, because they exist in the centres of the twin-type fracture mirror, as indicated by the arrows in Fig. 4b.

For the chemically-toughened specimens the indentation tests were made at further high indentation loads of 19.6 to 24.5 N at which the typical median crack is always produced for the as-received specimens. However, no case of the crack evolution was observed just after the indentation as well as at the lower load of 9.80 N. If the median crack starts from the bulk beneath the deformed zone, as considered by Lawn and Evans [16], the chemically-toughened specimens must show a median crack because the surface compressive layer of the chemically-toughened glass used in this study is considered to have little effect on the bulk beneath the deformed zone due to its extremely small thickness in comparison with the deformed zone depth of several tens of micrometres. However, median cracks were not observed in the chemically-toughened specimen, indicating that the median crack does not originally start from the bulk beneath the deformed zone. This

TABLE III 4-point bend test results of the indented specimens at 9.80 N in air at room temperature (20° C, 45% r.h.)

Specimen	Liquid nitrogen strength (MPa)
Chemically-toughened without cracks	703 ± 37* (9)†
Chemically-toughened with cracks	83, 87 (2)†
As-received	72 ± 2* (9)†

*Average ± 95% confidence limit.
†Number of measurements.

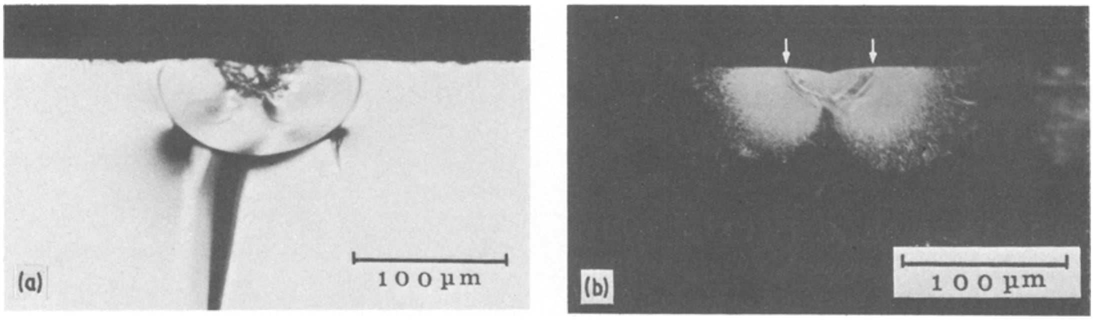


Figure 4 Fracture surfaces of the chemically-toughened glass (a) with and (b) without radial cracks. The arrows in (b) indicate both corners of Vickers impression at the surface. (Note: the fracture surface of (b) was obtained from the chemically-toughened specimen annealed at 500° C for 4 h, since the chemically-toughened specimen without the radial cracks showed very high strength and was broken into very small fragments. No crack healing occurs under the annealing treatment.)

result may, therefore, be considered to mean that median crack evolution also initiates from the surface.

Marshall and Lawn [13] have reported that the extension of the surface crack from the impression is reduced by the surface compressive layer and Fig. 4b suggests the existence of very small surface flaws at the corner of the impression. Therefore, it can be thought that some embryonic cracks are formed at the surface of the chemically-toughened specimen.

As to the possibility of embryonic initiations of the cracks at the surface during either the loading half-cycle or the unloading, Perrott [17] has presented a detailed stress field model derived from the elastic–plastic indentation for the point contact, which shows that a very shallow but high tensile stress field generates across the median plane in a surface region around the indenter. So, the crack nucleation in Vickers indentation possibly occurs in the near surface region around the indenter; however, it cannot fully extend into the surface because of the extremely small tensile zone during the loading half-cycle.

Fig. 5a shows the plane view of the Vickers impression with radial cracks of the chemically-toughened glass exhibiting the many minute cracks at the contact area between the indenter and the glass surface. It is known that surface cracks occur within the actual contact area of the indentation and are probably due to friction at the interface or high elastic surface stress [7, 18]. The surface stress can nucleate Hertzian-type ring, or segments of ring, cracks around Vickers indentations in soda-lime–silica glasses [7]. A view showing the corner of the impression at greater magnification (Fig. 5b), however, indicates that the radial cracks initiate from the cracks at the corner. This may be considered to be due to the stress concentration effects of displacement of material from orthogonal faces of the indenter. Thus, the corners of the surface of the impression provide some evidence for crack formations at the surface. In the case of the chemically-toughened glass, the high surface compressive stress may be considered to inhibit the instantaneous developments of the cracks just after the indentation.

From the embryonic crack initiations due to the Perrott stress field at the surface and the tentative no-crack developments in the chemically-toughened glass, it is believed that the crack initiations at the surface are essential for the formation of both radial and median cracks. That is, the embryonic crack initiations at the surface can be considered to determine the development of the radial and the median cracks resulting from the following features:

(a) minute cracks are produced at the surface contact area with the indenter;

(b) the minute cracks at the corner of the impression grow in depth along with the elastic–plastic boundary of the deformed zone beneath the indenter; and

(c) they are instantaneously developed by the tensile stress field built-up around the indentation area during or after the unloading (for the Types I and II arrest position marks, as illustrated in Fig. 3).

Also, the median crack development (the Type III arrest position mark) possesses both of the features (a) and (b), and the high tensile stress due to high indentation load at the elastic–plastic boundary makes the crack develop from the elastic–plastic boundary during the loading half-

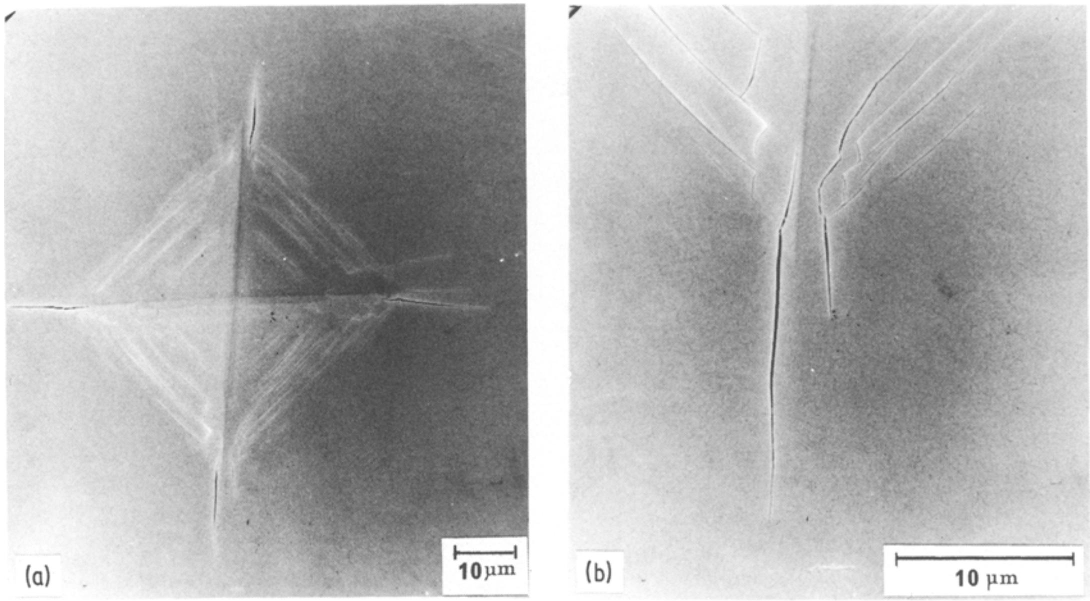


Figure 5 Scanning electron micrographs of Vickers impression with radial cracks (a) in the chemically-toughened surface and (b) a magnified view of the corner of the impression.

cycle. The subsequent arrest position marks, such as the 2nd, 3rd and 4th in Fig. 1f, are formed by the tensile stress built up around the indentation part after the unloading, since they tend to take semi-circular shape [8].

Thus, the cracks in Vickers indentations can always be considered to originate from the glass surface. The only difference between the radial and the median cracks is the timing of their developments in accordance with the magnitude of the indentation load: the radial crack develops after the unloading and the median crack develops during the loading half-cycle. It should be also noted that this conclusion is strongly supported by the existence of the Type II arrest mark with transient shape from the radial to the median crack which is formed during the unloading half-cycle at indentation loads intermediate between those for radial and median crack formations.

4. Summary and conclusions

The crack formation processes in soda-lime–silica glass were systematically investigated by changing the Vickers indentation load.

Three types of arrest position marks were observed in the crack surfaces, dependent on the magnitude of the applied indentation load, and the crack in annealed specimens, notwithstanding the shapes of the arrest position marks corresponding to the stress fields produced by the indentation. The crack development was considered to occur during the loading half-cycle and during or after the unloading at higher and at lower indentation loads, respectively.

Chemically-toughened specimens showed Vickers impressions without any cracks at indentation loads that are sufficient to create the median crack in annealed specimens, notwithstanding that the surface compressive layer was extemely thin compared with the depth of the deformed zone beneath the indenter, indicating that the cracks in Vickers indentation always initiate from the surface. The crack initiation at the glass surface was discussed on the basis of the Perrott stress field model and confirmed by scanning electron microscopy.

It is concluded that the embryonic formation of the surface crack in Vickers indentation is essential for both radial and median crack evolutions.

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Received 9 July
and accepted 19 October 1981