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Quantitative analysis of scratch-induced microabrasion on silica glass

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ABSTRACT

We employ instrumented nanoindentation for obtaining quantitative information on the onset of scratch-induced microabrasion on silica glass. For this, *in situ* evaluation of lateral force and friction coefficient is compared to *post mortem* optical inspection, following edge-forward scratching with a Berkovich indenter at velocities of 10–500 μm/s under continuously increasing normal load of up to 300 mN. In the two approaches, the onset of microabrasion is identified from the occurrence of pop-ins in the load-displacement curve and phenomenologically determined from the scratch pattern, respectively. Obtained data are analyzed in terms of a Weibull distribution, assuming that microabrasion sets-on as a result of acting stress as well as surface state. Aside of the occurrence of occasional outliers at low load (probably induced through individual surface defects), data indicate two underlying probability functions, *i.e.*, the probability for the propagating scratch to hit a surface flaw and the probability that such an event causes an observable micro-crack. Dominance of the former leads to an exponential function with Weibull modulus $\sim$1, reflecting a purely random distribution with load-independent probability of failure. This is observed in particular at high scratching velocity after passing a certain normal load. For the latter, the Weibull modulus increases with increasing scratching velocity, that is, from $\sim$1.6 to 4.4, at intermediate load. Here, low Weibull modulus at low load is attributed to the increasing time of local strain, which leads to a reduction of the load-dependence of micro-cracking relative to a faster-moving scratch. In the present case, the critical lateral load for microabrasion of silica (50th percentile) is around 30–40 mN. Within the employed experimental conditions, this value is practically independent of scratching velocity.

1. Introduction

As a commodity component in a variety of consumer applications, glass surfaces are frequently subjected to abrasive load and scratching. At the same time, most of these applications rely on the surface quality and visual appearance of the employed glass. Scratch-induced surface flaws may strongly compromise this aspect. In addition, they also act as stress amplifiers and, hence, reduce overall mechanical performance. Understanding abrasive damage and the underlying material properties has therefore been a subject of significant interest. However, present considerations of the scratch and abrasion resistance of glasses are mostly phenomenological [1–5]. In particular, this concerns the established protocols for scratch testing, which provide only qualitative information. In the first steps towards a rigorous and eventually mechanistic description of the scratching process on glass surfaces [1], Le Houérou considered the archetype example of soda lime silicate glass by dynamic micro-indentation. Applying an increasing normal load on a Vickers indenter during linear lateral displacement at constant speed, they found a characteristic pattern in which distinct regimes of damage are observed. With increasing normal load, these comprise, in sequence, of plastic deformation, micro-cracking, chipping and micro-abrasion, Fig. 1a–b.

According to these early observations, radial (chevron) cracks are the first flaws which appear beyond the ductile regime, at relatively low load. With increasing load, lateral and median cracks reach the surface and form chips. Here, median cracks propagate deeply into the material while lateral cracks appear close to the surface in a depth which lies within the so-called plastic zone [6,1]. A broad variety of parameters determines this phenomenology, including the rate of scratching, the indenter geometry relative to the scratching direction, the applied normal force, glass surface conditions, environmental atmosphere and humidity, and the presence of debris or impurities on the specimen surface. So far, the concrete action of these parameters has received only very limited attention [2,7,8]. This is particularly the case for the technically relevant question of compositional dependence. Here, recent approaches to compositional development rely largely on the assumption that data obtained from normal indentation correlate directly (or even linearly) with damage resistance under lateral contact

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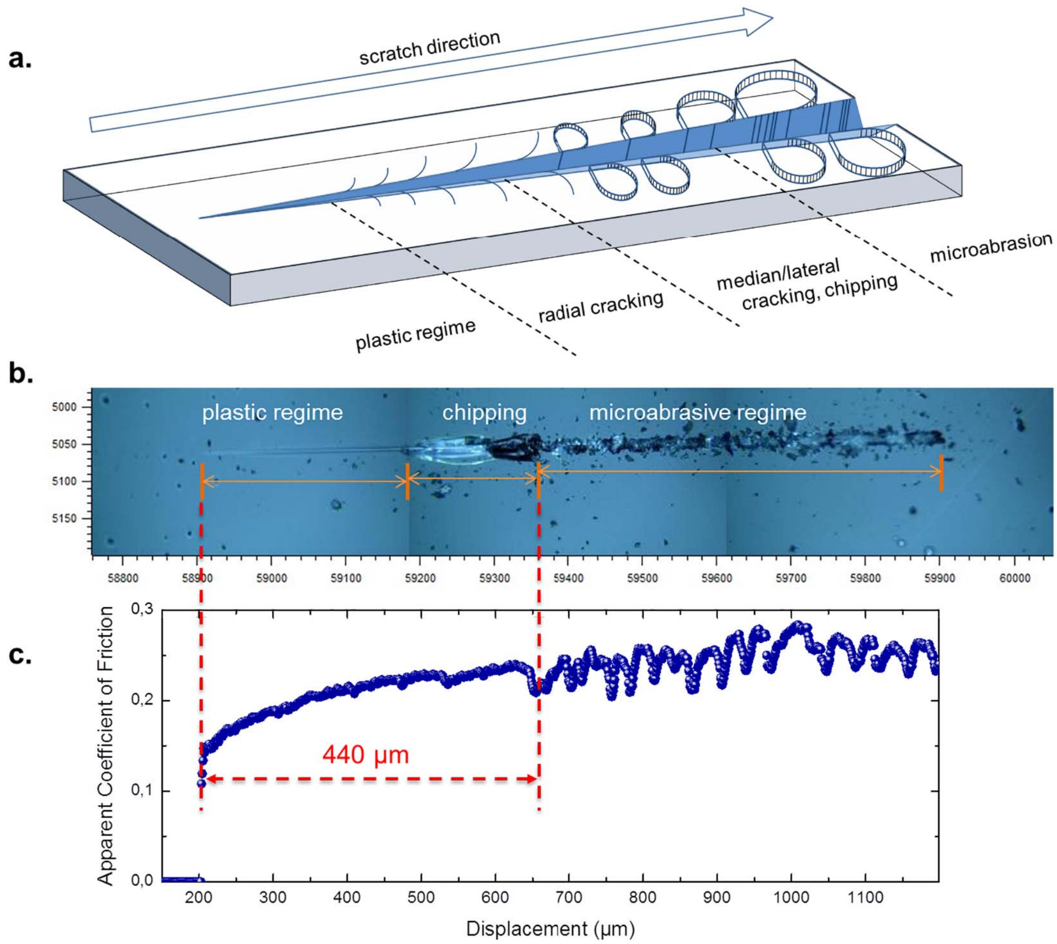

Fig. 1. Typical scratch pattern which is observed on silicate glasses during steady scratching with increasing normal load (a). In (b), this phenomenology is shown for the specific case of fused silica at a scratching rate of 50 μm/s and a normal load which increases from zero to 300 mN, using an irregular diamond edge for scratching. (c) is a representation of the corresponding variation in the apparent friction coefficient (see text for details).

load. The consensus is that the indentation response of glasses is governed by the interplay of elastic deformation, structural compaction and shear [9]. Relaxation studies can subsequently be used to evaluate individual contributions of the latter two [10–12]. Then, the ability of the considered material to compact depends directly on its free volume, on molecular scale, and correlates with Poisson's ratio [13]. Accordingly, vitreous silica, with exceptionally low Poisson ratio and high free volume, exhibits a degree of structural compressibility which beats that of almost all other glasses. However, it has also become clear that the structural reactions which underlie damage infliction are significantly more complex [14].

In the present letter, we report on lateral force analyses during scratching of vitreous silica in an effort to obtain increasingly quantitative information on the scratch resistance of glasses. For this, we consider the effect of scratching velocity (loading rate) on the onset of micro-cracking and chipping. The correlation of *in situ* recordings of friction forces with *post mortem* imaging of the scratch enables the identification of onset points for scratch-induced fracture events and microabrasion. This is subsequently evaluated through Weibull statistics.

2. Experimental

2.1. Scratch testing

Instrumented nanoindentation (G200, Agilent) was employed to generate quantitative data on the scratch resistance of commercial-grade vitreous silica (Heraeus Suprasil 1). The experiment comprises control of the normal load L_N on a Berkovich tip and recording of the lateral load L_L during lateral displacement as illustrated in Fig. 2. The value of L_L results from a specific rate of normal loading and lateral displacement. It is determined from the lateral stiffness of the indenter, K_L, and its displacement in *x*- and *y*-directions, shown schematically in Fig. 2a. The overall observation length (lateral displacement) was kept constant among all samples (1.0 mm). Samples themselves were cylindrical with a diameter of 33 mm and thickness of approximately 3 mm. On the studied surface, they were sequentially polished with dry silicon carbide powder with grain sizes of 70, 40 and 9 μm, and finally with a suspension of diamond powder with a grain size of 1.0 μm, leading to an average roughness of 1.19 μm (mean arithmetic height, taken from confocal microscopy) and subsequently stored in vacuum. Directly before analysis, the samples were cleaned in an ultrasonic bath of pure isopropanol for 5 min at room temperature, and subsequently flushed with ethanol. Tests were conducted by increasing the normal load L_N from 0.05 mN to 300 mN during lateral displacement at rates of 10, 50, 100, 150, 300 and 500 μm/s across the overall lateral displacement range of 1.0 mm, at room temperature. This corresponds to normal loading rates between 3 mN/s and 150 mN/s. The employed tip geometry is shown in Fig. 2c. Scanning was conducted in edge-forward configuration (EF, Fig. 2b). For each test, an initial specimen surface profile was obtained before scratching by pre-scanning the sample's surface with the indenter under a load of 50 μN. While testing, both the penetration depth and the value of L_L were continuously monitored. After scratching, the surface profile of the sample was scanned again under the same conditions as during the pre-scanning stage. For each loading rate, 20 scratches were performed. Scratch