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Investigation of wear mechanisms through in situ observation during microscratching inside the scanning electron microscope

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Abstract

Scratch experiments have been used in the past to investigate various contact phenomena such as abrasion mechanisms or asperity contacts. Depending on tip geometries, loading conditions and materials investigated, different crack propagation modes (radial or lateral cracks, etc.) or deformation modes (ploughing, chipping, etc.) may dominate the scratching process. The residual scratch path can yield some information about dominant deformation and fracture modes. It is, however, often not possible to uniquely correlate cracks and other phenomena with events on the recorded load–displacement curves.

We have built a miniaturized microscratch device for use inside a scanning electron microscope (SEM) that allows the observation of the surface around the tip with sub-micrometer resolution during scratching. Using a conical indenter with spherical tip we demonstrate on different materials that the device is a powerful tool to observe initiation and propagation of cracks, to observe the flow of the material near the indenter (piling-up and sinking-in) and to study chip and particle formation mechanisms during microscratching. In GaAs, particles were observed to form in front and on the rear side of the tip via interaction of chevron cracks. In the case of a Fe-based bulk metallic glass, shear bands were observed to form in front of the tip leading to serrated chip formation. Discontinuities in the tip penetration during scratching of a polymer thin film were related to the onset of crack formation behind the tip and to the propagation of semi-circular cracks in front of the tip. The observed large elastic recovery of the polymer film at the rear side of the tip has to be taken into account for accurate contact area calculations.

Keywords: Microscratching; In situ observation; Bulk metallic glass; Polymer thin films; GaAs

1. Introduction

Scratch resistance of materials is of importance in many technological applications such as automotive clear coats or glasses. In a wider sense, scratch testing serves as (a) model experiment to understand deformation and crack formation mechanisms in precision machining and (b) is fundamental to the understanding of deformation and removal mechanisms in grinding, polishing and abrasive wear, because all these processes are functions of the cumulative actions of repeated microscopic contacts. Scratching is also used as a test procedure to determine qualitatively the mechanical properties of coatings, in particular the coating-substrate adhesion [1].

In *ductile bulk materials*, the behaviour of the material during a scratch test depends mainly on the shape of the indenter that controls the imposed deformation level and on the mechanical properties of the material. Bucaille et al. showed that during scratching the average deformation is related to the ratio of the penetration h of the indenter to the contact radius a [2]:

$$\varepsilon_r \propto \frac{h}{a}. \quad (1)$$

For a conical indenter having a spherical tip, the deformation level h/a increases as the penetration of the indenter

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increases. For small deformations, the behaviour of the material is mainly elastic and no permanent groove can be observed after scratching. If the deformation imposed by the indenter increases, ductile ploughing is observed [3,4]. The transition between elastic and plastic domains also depends on the rheology of the scratched material. For an elastic-perfectly plastic behaviour, the flow of the material near the indenter is strongly dependent on the ratio between Young's modulus, E, and yield stress, σ_y [5]. For high values of E/σ_y pile-ups are created near the indenter, while for low values of E/σ_y the material sinks-in in front of the indenter and elastic recovery at the rear of the indenter is large [2]. This means that for polymers, i.e. materials having a large elastic part ($E/\sigma_y \sim 10$–50), the deformation mode can vary from the elastic behaviour with almost no residual groove to ductile ploughing. For metals ($E/\sigma_y > 200$), even for a small deformation level, ductile ploughing is the main observed mode, followed by chip formation as the deformation imposed by the indenter increases.

In *brittle solids*, a large variety of cracks and deformation phenomena can be observed [6,7]. Soda-lime glass is an extensively studied brittle model material: Swain has carried out a detailed study using Vickers tips already in 1979 [8]. His observations of the morphology of the scratch path and of the extent of cracks below the surface after scribing can be summarised as follows: at loads below 0.05 N, there is no visible surface or sub-surface cracking around or within the residual groove. Well-developed median and lateral cracks are visible at intermediate loads (0.1–5 N), with lateral cracks not intersecting the surface at loads up to 1 N. At high loads, median cracks and poorly developed lateral cracks are observed and the region around the scratch track is seen to have a "crushed" appearance. Similar observations have also been reported by Ahn [9]. Bulsara investigated in situ the contact region between a Vickers tip and soda-lime glass using an optical microscope looking from the back side [10]. He observed that depending on the applied load median and lateral cracks are generated either during unloading behind the indenter or along with the sliding tip. Radial/chevron cracks were found to arise due to median-type cracks forming ahead of the sliding indenter and deviating to either side of the scratch track. Due to the geometrical self-similarity, the deformation imposed by the indenter and defined by Eq. (1) is constant for Vickers or conical indenters as the penetration of the indenter increases. The transition between these deformation modes can therefore not be related to a variation of the deformation imposed by the indenter. However, the volume solicited by the scratching process increases as the penetration depth increases which allows the activation of more defects which initiates brittle fracture.

In this work, the deformation and fracture phenomena occurring during microscratching with a rounded conical indenter are studied using an in situ observation technique inside a scanning electron microscope (SEM). The electron microscope allows imaging a major part of the contact region with sub-micron resolution during microscratching. There have been already excellent studies on deformation and fracture modes using scratch techniques inside an SEM [11,12]. For instance Hedenqvist and Hogmark revealed fracture modes of TiN coatings on steel during scratching [11,13]. Our experimental set-up differs significantly from approaches in the past, as we use piezo-based actuators that allow scratches with nanometer displacement resolution. In order to explore the potential of the technique, scratch tests were performed on a semiconductor, on a metallic glass and on a polymer thin film sample. Furthermore, the results of the in situ SEM measurements are compared to scratch results from a commercial nanoscratch instrument.

2. Experimental

2.1. Samples

Materials belonging to several different categories in terms of mechanical properties were investigated to explore the potential of the SEM-based microscratch device: a Fe-based amorphous metal (also known as bulk metallic glasses) sample, single crystalline GaAs and a polymer thin film. The Fe-based bulk metallic glass sample has a nominal composition of $Fe_{61}Zr_8Y_2Co_5Cr_2Mo_7B_{15}$ and was produced by arc melting and drop cast into a copper mold. The specimen was mechanically polished to a mirror finish. More details on the processing and full characterization of the microstructure can be found in [14]. The GaAs wafers were of (0 0 1)-type and not doped. The polymer thin film was a 5 μm thick polysiloxane coating containing 20 vol.% colloidal SiO_2 particles on a thermosetting-based polycarbonate substrate. Such types of coatings are applied for eyeglass lenses. The material behaviour during a scratch is therefore of direct interest in the application of this type of coatings. For more details see Bucaille et al. [2].

2.2. Experimental set-up

2.2.1. Conventional microscratching

A Nanoindenter-XP (MTS/Nanoinstruments, Oak Ridge, TN) equipped with a lateral force measurement option for scratch experiments was used to determine the indentation hardness and the Young's modulus of the investigated samples and to perform scratch experiments with a linearly increasing load from 0 to 300 mN. A conical diamond indenter (45° half-angle) with a spherical tip having a tip radius of around 1 μm was used for the scratches. The scratch length was 500 μm and the scratch velocity was 10 μm/s. Lateral force and penetration depth were continuously recorded as a function of the applied normal load. For each sample several scratches have been made to guarantee the unambiguousness of the obtained results. For each scratch the specimen's surface profile has been measured prior and after the scratch using the same conical indentation tip with a constant load of 50 μN. The GaAs(0 0 1) surface was scratched along the