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Implications of Polishing Techniques in Quantitative X-Ray Microanalysis

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Specimen preparation using abrasives results in surface and subsurface mechanical (stresses, strains), geometrical (roughness), chemical (contaminants, reaction products) and physical modifications (structure, texture, lattice defects). The mechanisms involved in polishing with abrasives are presented to illustrate the effects of surface topography, surface and subsurface composition and induced lattice defects on the accuracy of quantitative x-ray microanalysis of mineral materials with the electron probe microanalyzer (EPMA).

Key words: abrasive wear; bound abrasives; chemical-mechanical polishing; loose abrasives; polishing; surface and subsurface damage; x-ray microanalysis.

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Contents

1. Introduction

An optically flat polished surface is a necessary criterion satisfying the geometrical conditions for quantitative x-ray microanalysis with the electron probe microanalyzer (EPMA). All mechanical, structural, physical and chemical surface modifications resulting from the surface preparation will affect the accuracy of quantitative x-ray microanalysis as previously reported by Rémond [1]. The objective of this presentation is to convey to the EPMA community that polishing with abrasive particles is a complex operation involving many experimental and instrumental factors that are characteristic of the materials to be polished. For this purpose, the mechanisms involved in abrasive wear will be presented in order to illustrate some consequences of the polishing procedure on the reliability of quantitative x-ray microanalyses.

Mechanical polishing is performed by means of abrasives with decreasing grain size until scratches are no longer visible (optically polished surface). From a mechanical point of view, during the first stage of preparation, coarse grains are used to remove initial surface topographical and chemical defects. The next stage with smaller abrasive grain size aims to obtain the final quality of the surface satisfying the conditions for EPMA analysis. This regime is often divided into two operations, e.g., the intermediate polishing and the final polishing.

The mechanisms involved in mechanical polishing using abrasive particles are part of tribology, the discipline studying material science, physics, chemistry and surface contact engineering [2-5]. A description of a tribological system (according the norm DIN 50 320) consists of a set of experimental parameters (applied load, velocity and duration of the motion) and the system structure (the two bodies in contact, the interfacial and surrounding media), as shown in Fig. 1.

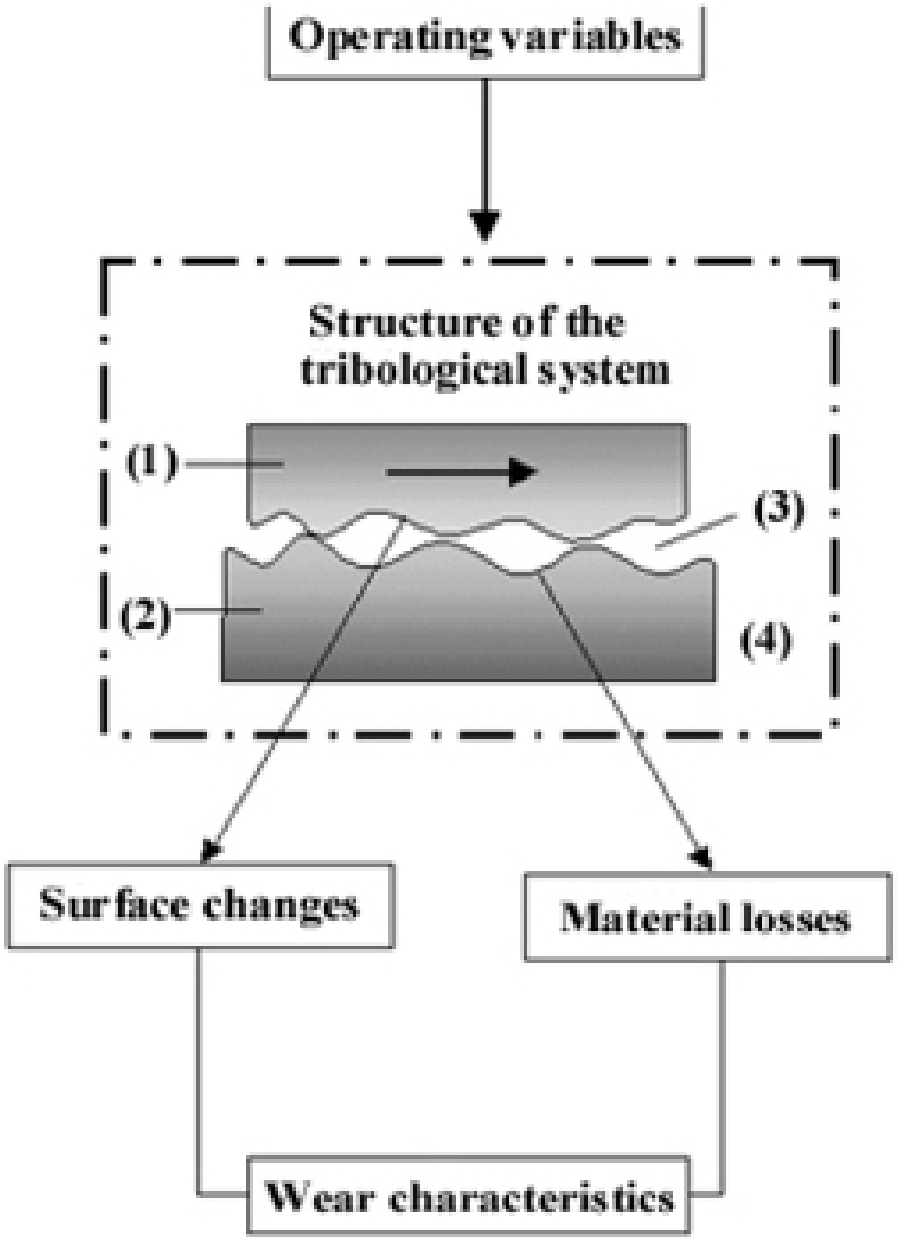

Fig. 1. Schematic representation of a tribological system according to the norm DIN 50 320. The tribological system consists in (1) the specimen to be polished, (2) the abrasive specimen, (3) the interfacial medium and (4) the surrounding medium.

Wear is defined as a cumulative surface damage phenomenon in which material is removed from a body as small debris particles, primarily by mechanical processes. The wear mechanism is the transfer of energy with removal or displacement of material. The four major wear mechanisms are adhesion, abrasion, surface fatigue and tribochemical reactions. In polishing with abrasive particles, the wear mechanism is mostly abrasive wear but other mechanisms are also possible. The abrasive mechanisms occurring in a dry or humid environment, result from the simultaneous actions of normal and tangential forces and are materialized by the development of ploughing grooves or scratches which are in some instances accompanied by hertzian fractures. For the classification of the abrasive wear modes, we will use the most widely accepted terminology known as two-body abrasion and three-body abrasion. This terminology illustrates the experimental situations encountered in the polishing techniques as illustrated in Fig. 2. In a two-body mode, the bound abrasive particle (identified as a guided-cutting tool) is solidly fixed to the substrate (Fig. 2a). In a three body

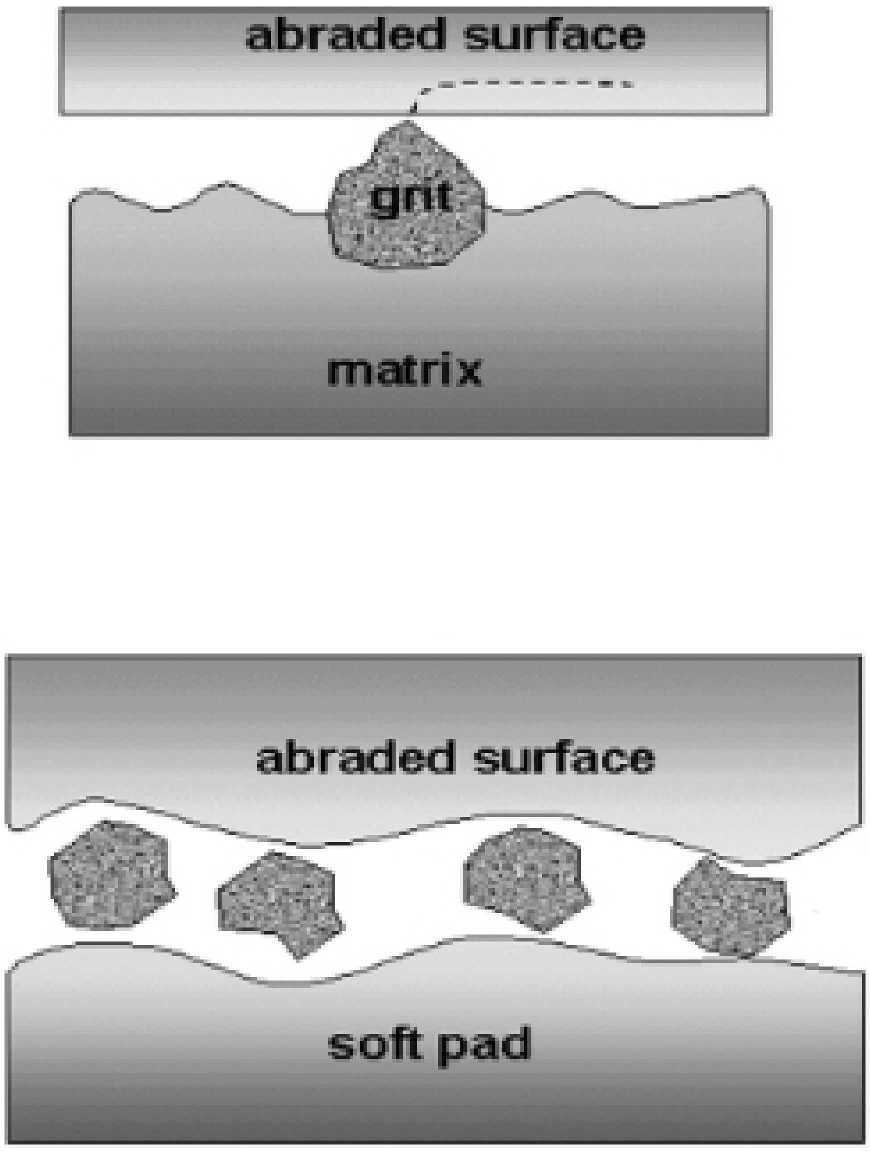

Fig. 2. Abrasive wear in two body and three body configurations. a) two body situation with abrasives bound to the polishing substrate, b) three body situation resulting from wear debris or loose abrasives trapped in the interface between the specimen and a polishing pad.

abrasive mode, free (or loose) particles form a slurry between the specimen surface to be polished and a flat polishing substrate as illustrated in Fig. 2b. The free particles in a three-body wear mode may be intentionally added abrasives or be detached debris from the worn surface.

The manifestations of abrasive wear are the change of the surface roughness resulting from material removal and the change of the physical and chemical properties of the surface and subsurface with respect to those of the bulk. In addition to the mechanical and geometrical description, these deformations are accompanied by (i) the production of highly localized heat, (ii) the creation of excitations and defects in the material, (iii) the production of dangling bonds and trapped electrons, (iv) and the emission of excited and reactive species into the gas phase (exo-emission). All these phenomena result in a highly reactive surface accompanied by the formation of a surface composition different from that of the bulk. Separation of charges also leads to the creation of intense electric fields at the surface of many insulating materials.

When a single type of material is routinely analyzed with the EPMA, it is possible to optimize the polishing strategy, minimizing the thickness of the damaged surface. However, there is no general selection rule for the operating conditions because, for a given material, the wear mechanisms depend not only on the specimen properties but also on all of the tribological interactions between the abrasive materials and the specimen to be worn (see Fig. 1). Generally, most of the EPMA laboratories have to analyze a variety of specimens which often contain several different phases. Standards blocks also contain several materials and the polishing procedure cannot be optimized for all phases present in the heterogeneous material.

The consequences of the abrasive wear on the accuracy of quantitative x-ray microanalysis data are illustrated by the following examples: (1) chalcopyrite ($CuFeS_2$) in a massive form and as inclusions in a silver sulfide matrix, (2) a binary quartz (SiO_2)-arsenopyrite (AsFeS) mineral, (3) α-alumina crystals and (4) synthetic polycrystalline ZnS obtained by chemical vapor deposition.

2. Abrasive Wear

2.1 Two Body Abrasive Wear

Models of abrasive wear with bound abrasives assume that the abrasive asperity is like a sharp tool producing a groove into a surface (Fig.3).

The volume dV of material removed by an individual rigid cone shaped punch with a half-apex angle θ sliding at the surface of the specimen along a distance dL under an applied load dP is:

$$\frac{dV}{dL} = \frac{2\cot g\theta}{\pi}\cdot\frac{dP}{H} = \frac{2\tan\alpha}{\pi}\cdot\frac{dP}{H} \qquad (1)$$

where H is the Meyer hardness of the indented material and α is the attack angle of the particle for which the sliding direction is parallel to the specimen surface.

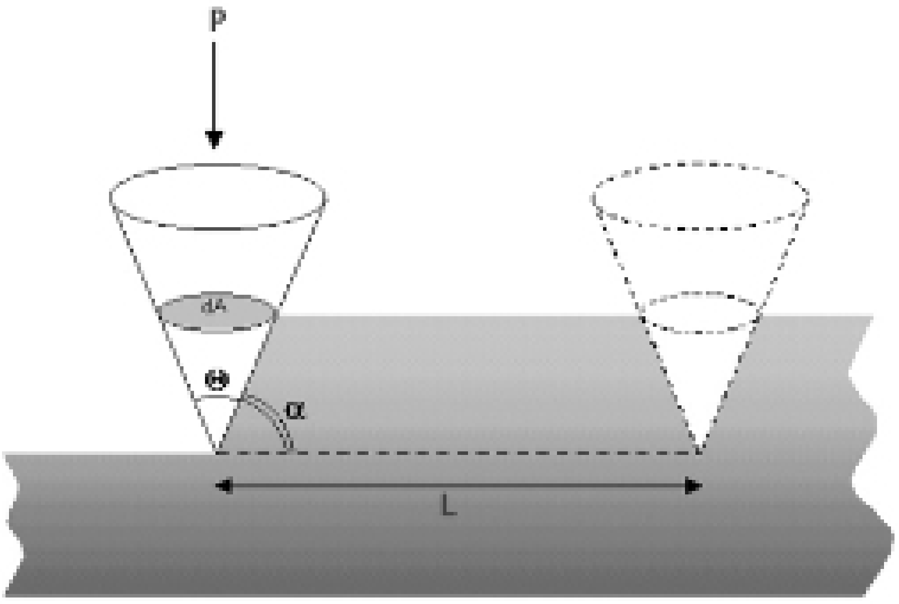

Fig. 3. Model of abrasive wear by a conical shaped particle.