ABSTRACT

During preparation of microstructure, often referred to as cutting, mounting, grinding and polishing, the structure may be influenced to such a degree, that the subsequent examination in the microscope can show incorrect results. In general, it is believed, that as long as the surface appears shiny and reflective without scratches, the preparation is OK, but the real life shows, that this may not always be correct. This paper describes an alternative approach.

Key words: Damage, methods, reproducibility.

1. INTRODUCTION.

1.1 History

Metallographic preparation came out of industrial processes and thus the use of emery paper grinding and Al₂O₃ polishing on soft cloth to ensure a shiny, reflective surface (just as in the industry) became the standard way to prepare sample. Scratchs were related to deformation, which at that time were seen as the only (main) type of problem to over come during preparation.

In the middle of the 50', it was detected, that e.g. the carbon content in cast iron measured in a micro-structure was very different from the actual content. Which lead to the detection of the pull out problem, whereby the carbon was pulled out leaving holes much larger than the original carbon-phase, see figures below. This explained the difference, and the consequence was introduction of diamond polishing, which were able to solve this problem.

Since then, it has been realised, that sample preparation has to deal with a variety of damages (or artifacts) caused by the preparation, and that a shiny, reflective surface (without scratchs) were no guarantee for a real structure or even, that the deformation had all been removed.

2. THE ALTERNATIVE APPROACH

Realising, that preparation shall remove damages caused by the processes itself (see next Fig.2), it is a must to understand, what
these damages can be, what they look like and how we can minimize their occurrence.

**Fig.2 Variety damages of sample.**

If we are able to visually identify the damages introduced from e.g. cutting, we can follow step by step through the processes, how these damages gradually are being removed. When they are completely removed, it must be anticipated, that the structure so obtained, are real.

Preparation, by which the operator have to follow the development of the structure both between each step and within one individual sequence is naturally time consuming, but there doesn't seem to be any better alternative. Oppositely, if the process can be controlled and repeated, the successive approach only needs to be done once. If all parameters like time, pressure, abrasive type, size, quantity etc. can be repeated (using controlled machines), this approach to reach the real structure is no more time consuming than any other methods, on the contrary.

Finally, it must be considered, that depending on the characteristic of the material- it's machinability in terms of hardness and ductility/brittleness- various types of consumer items and combinations of these, should be selected. Likewise, a variety of steps may be chosen depending on the materials properties. To use SiC grinding paper #220, 320, 500 and 1000 was made a routine for steel back in the early 50's, but it doesn't mean, that this approach is right for all materials (except for more than 1000 HV) on the contrary. For very soft aluminium, for instance, SiC paper #500, 1200 and 4000 may be a better procedure, and for steel today, we might use a special disc like Petrodisc-M in stead of SiC paper. And to use SiC paper for hard, high-tech ceramics, may produce a shiny surface, but it will seldom produce the real porosity.

To adjust selection of consumer articles, steps and process parameters to a certain material, is called to make a method for that material or sample. Modern sample preparation machines has computer controls, in which these methods can be stored for repeating sample preparation ensuring integrity. And particularly in R & D, such machines should always be used inorder to ensure, that e.g. samples from a trial series, can be compared.

3. DAMAGES.

We may divide the types of damages, which we introduce during cutting in particular, in 3 groups:

1. Deformation
2. Removals
3. Additions

Deformations occurs especially in softer materials. Atomic level deformations, like dislocaiton and twin, may not necessarily be visible in the optical microscope, and thus we may not have to remove them completely, however, as seen **Fig.3**, twins introduced from prepartaion may easily mix with deformations twin present in the structure, and thus present a problem during examination, as there is no visible difference between the two.
Deformations of the grains, often referred to as "smearing", is never acceptable, and can cause problems when examining soft layers: e.g. gold, copper, silver etc. on a hard base or on ductile materials with porosities, like super-alloys, cast steels and Ti, Zr, Ni-alloys, PM's and metal base spray coatings.

Removals are normally pull-outs of phases, inclusions (partly or fully) or whole grains: e.g. brittle and sintered materials. The effect is that a hole will be visible instead of the element pulled out, but both often being black in colour, it can be difficult to see any difference. Pit is pull outs of very small elements, often in "pure" metals, and in percent, the error to evaluate the size becomes larger, the smaller the elements are.

Pull outs are giving problems in most metals' examination and particularly in ceramics, where pull outs looks identical to real porosities, and this can lead to rather incorrect measurements of a structure's porosity. In ceramic coatings, where the real porosity is difficult to determine by means of gravimetric measurements, this problem is worse. (See Fig. 4.)

Additions cover a variety of problems including scratches and edge rounding. Both of these are easily recognisable, however, exactly because they are easy to detect, they don't present major problems when setting up a method for obtaining the true structure. The deformations underneath a scratch may sometimes cause problems, but not the mere fact, that there is a visible scratch. It is easy to remove and we can easily see, when the problem has gone.

Fig. 3 Twin in the copper after cutting procedure.

Fig. 4 Pull-out and Scratch Problem in Brass.

Edge rounding around pores present a larger problem, especially if it is combined with pull outs. Above brass structure shows, how different it can be, when elements are pulled out and the edges of the remaining holes are rounded.

4. BASIC REMEDIES

Apart from minimizing the damage originally introduced by selecting appropriate cut-off wheels and machines and non-damaging mounting method, 3 important factors has proven indispensable:

1. Using sharp abrasives for grinding and polishing.
2. Selecting hard backings (grinding and polishin papers/discs/cloth)
3. Dividing grinding and polishing into PG (plane grinding), FG (Fine grinding), DP (Diamond polishing) and OP (Oxide polishing).
The sharp abrasives ensure a minimum of deformations (especially smearing) and pull outs. Most appropriate abrasives are SiC and diamond-, less appropriate are oxides (like alumina).

Hard backings ensure the required flatness minimizing edge rounding, relief, comet-tails and expression grains.

Dividing into the mentioned 4 stages allows all important matters to be controlled; PG ensures a uniform damaged layer; FG an appropriate fast removal of the major part of this layer; DP removes damages related to smearing, removals and additions; and OP, using a chemical / mechanical method based on OP-S or OP-U with 0.04 micron abrasive particles, ensures removal of ever atom level deformations and final scratches.1

If these 3 points are observed, and controlled machinery as shown Fig.5 are used to work out appropriate methods, it is possible to obtain true structure, which then in turn are easily etched (including colour above mentioned alternative approach, and the fact, that more and more metallographic labs are turning in this direction indicate the correctness of this philosophy.

More and more effort are put onto preparation and equipment, including training of operators in order to assure that what is examined in the microscope is real, and not some cracks, boils or impressed elements, which was created during the preparation.

REFERENCE


PROFILE

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Graduation of Copenhagen University (Denmark) as Master degree of Mechanical Science in 1974 and dedicating to the metallography in Asia (Japan, China, Taiwan and Korea) through the practical metallographic seminar.

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