USE OF LUCIA IMAGE ANALYSIS IN MATERIALS STUDY

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The present study is concerned with the utilization of image analysis in materials research. Some utilization possibilities of image analysis in efficient acquiring of information on materials are demonstrated on examples of silicate and polymeric materials, a liquid-oil mixture and on carbonation of $Mg(OH)_2$. Image analysis and the possibilities of computer processing are highly valuable tools also in teaching where they allow the subject matter to be vividly presented with the use of the demonstrating capabilities of the image analyzer employed.

INTRODUCTION

Materials research is inseparably associated with evaluation of microstructure, texture, phase composition, porosity and additional parameters permitting not only the behaviour of materials to be explained, but also their behaviour under other conditions to be predicted and various technological measures to be proposed for their manufacture so as to improve their performance properties. The present contribution has the aim to demonstrate on examples the wide variety of possible uses for image analysis in the study of not only silicate materials.

At the Department of Glass and Ceramics (ÚSK), work with utilization of LUCIA image analysis has already been under way for three years. Just as the company Laboratory Imaging s.r.o. has been successfui in the development of new systems, ÚSK was modernising its equipment (the originally black-and-white system was replaced by a colour one). Although at first the B&W system was thought to be quite satisfactory for the pur-poses envisaged, the steadily growing numbers of people interested in solving their problems with image analysis showed that use of both systems was unavoidable.

Use of the image analysis (IA)

The IA apparatus at the disposal of ÚSK is of quite a high standard and has been successfully used in solving a variety of problems also for other departments of the Faculty of Chemical Technology. The components of the apparatus can be divided into several groups according to their function:

Image analyzers
- LUCIA D (B&W system with 256 levels of grey)
- LUCIA M (colour system with 16 million colours and 16 levels of grey)
- LUCIA G (B&W and colour system with 16 million colours and 256 levels of grey).

The images can be scanned in several ways:

1. with a camera - used to obtain macroimages, micrographs from optical microscopes, micrographs from electron microscopes, etc.,
2. with a scanner,
3. from a videorecording.

The camera works with a resolving power of 800×600 pixels, and the image information from a videorecording has a resolving power of 640×480 pixels. Selection of the equipment then depends on the required resolving power of the images.

Several media can be used for storing the images. In our system there are the following:

1. Storing on a 3.5" diskette: however, the method has the great disadvantage in that the diskette has the capacity for only one colour image or two black-and-white ones.
2. Storing on the computer hard disk - limited disk capacity is likewise a drawback.
3. Storing on other disk media - in our case, use is made of SyQuest disks of 270 MB capacity.
4. Storing on a videotape - there is the disadvantage of poorer resolving power (640×480 pixels).

Printing of the images and evaluated data can be effected with the use of various types of printers (in our case HP 520 (550), EPSON Stylus Color, laser printers) or with that of videoprinters (in our case MITSUBISHI CP 53 E) which produce high-grade images but only of a fixed size.

Communication possibilities are provided by connecting the computer to Internet, which also greatly facilitates transmission of images between individual laboratories.

Application in the field of ceramics

The properties of ceramic materials are greatly influenced by their **phase composition.** Examples of the appropriate applications are given in the following figures.

Figure 1 depicts grog (quartz, feldspar) in a ceramic body. The content of grog and its particle size distribution are important parameters when characterizing the properties of ceramic products (optical microscope, crossed nicols).

Figure 2 shows dispersion of ZrO_2 particles in a matrix of Al_2O_3. This type of so-called transformation-hardened ceramics belongs among prospective hi-tech materials of high strength. In this case it is important to know the size distribution of ZrO_2 particles and possible existence of their clusters (electron microscope, reflected electrons).

Information on the orientation of particles in a ceramic matrix is also of considerable significance. As an example, we present the microstructure of an ancient ceramic material, where the orientation of particles allows the progress in manufacturing technology (forming method, firing temperature) to be assessed, and clues indicating the origin of the objects to be estimated. Figure 3 shows a microscopic polished section of a vessel found in Germany in the region of the town Cham, the site of the so-called Cham Culture.

Strength evaluation is of primary importance in many fields of materials research. Strength depends above all on the condition of the surface and that of the surface layers. The presence of fissures and cracks results in inadequate strength. To describe the behaviour of products under load one has to know the length and width of the fissures and cracks, their orientation with respect to the surface, etc. This information can be easily obtained by means of image analysis. In this case, obvious advantages are also provided by the possibility of readily archiving and demonstrating the defects revealed. Examples of such defects in stoneware tubes are shown in figure 4. The samples are polished sections examined in incident light. The so-called fracture toughness of materials (K_{Ic}) is tested by measuring their bending strength on samples with an artificially prepared notch (crack) in the specimen surface. The notch length and width must then be introduced into the calculation. The values can again be conveniently determined and archived by the LUCIA system. An example of such a notch (crack) is given in figure 5.

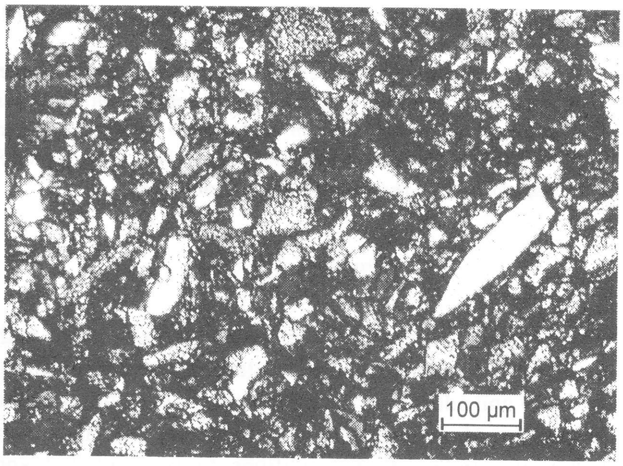


Figure 1. A Grog in a ceramic body.

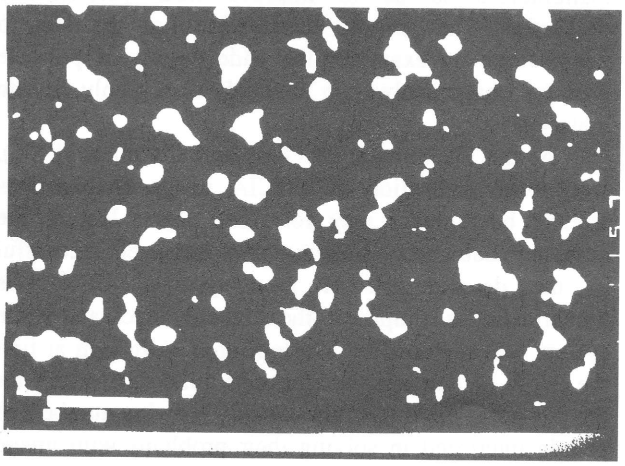


Figure 2. Dispersion of ZrO_2 particles in a matrix of Al_2O_3, the bar in the figure is 10 μm in length. An example of evaluation printout is also given below.

Number of Fields	1			
Number of Objects	144			
Objects per Field	144			
Measured Area	1453.2 [μm*μm]			
Objects per Area	0.0990918 /[μm*μm]			
Area Fraction	0.144531			
Feature	**Mean**	**St.Dev**	**Minimum**	**Maximum**
Area	1.4586	1.6994	0.01265	11.102
EqDiameter	1.175	0.69027	0.12691	3.7598 μm
Perimeter	4.0609	2.5287	0.38431	14.473
Circularity	0.851	0.11569	0.29734	1

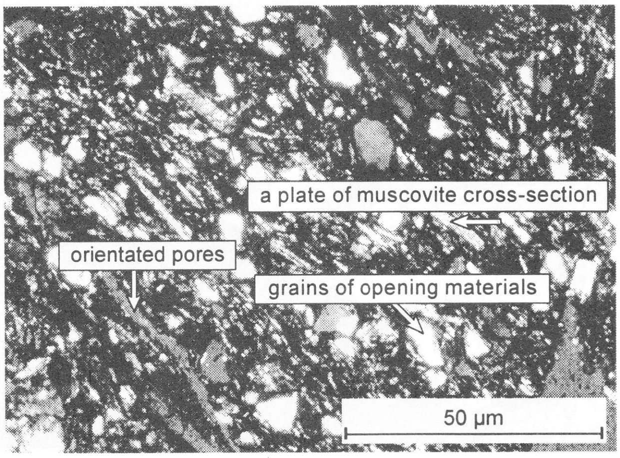


Figure 3. Microstructure of ancient ceramics (Cham Culture).

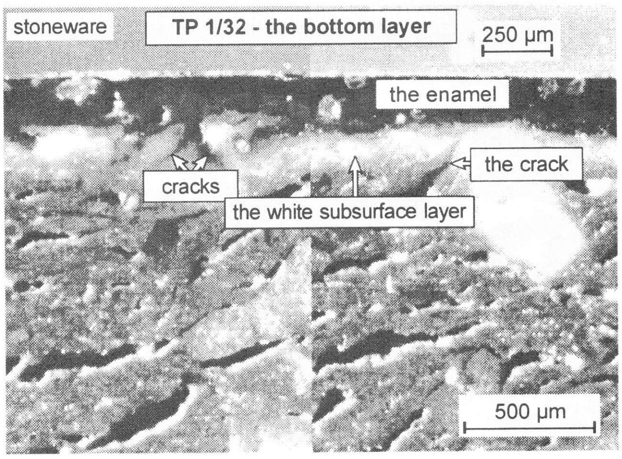


Figure 4. Defects in the surface of stoneware tubes.

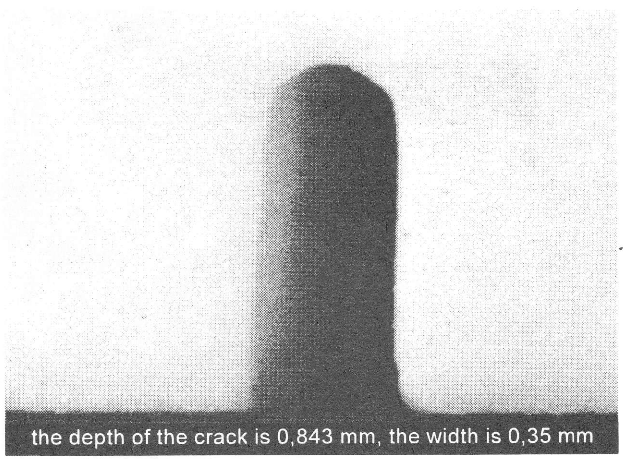
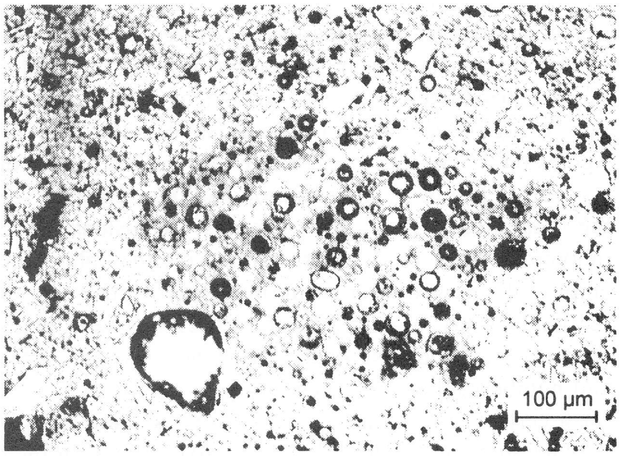
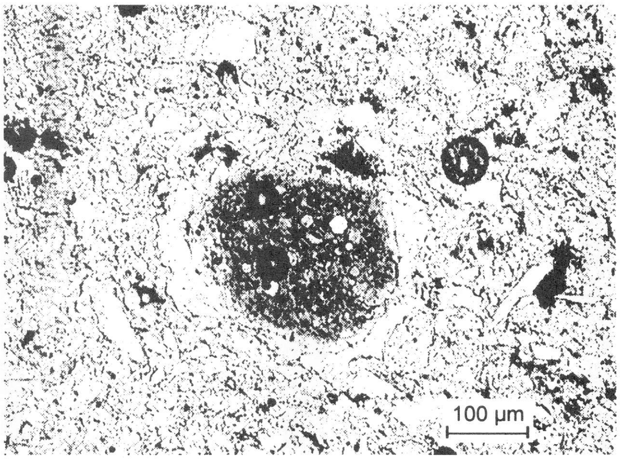


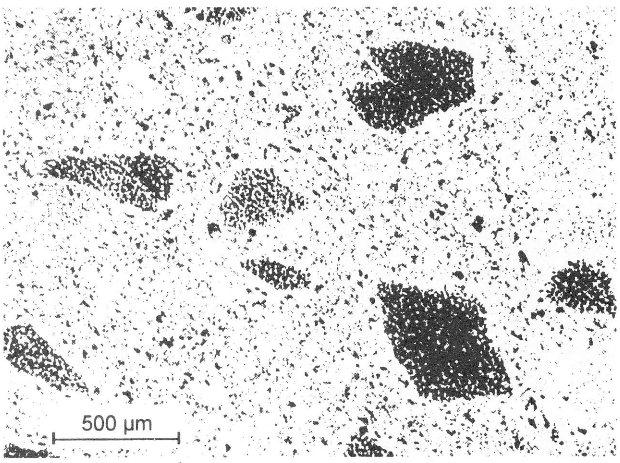
Figure 5. Artificially cut crack (notch) in the surface of a ceramic specimen.



a)



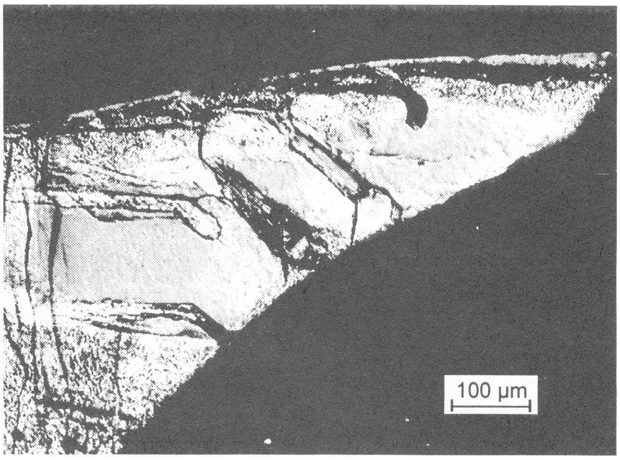
b)



c)

Figure 6.
a) - Flux mix A 42; *b)* - Flux mix B 42; *c)* - Fired phonolite Želenice Z 33

The resultant properties of ceramic products depend to a considerable degree on the raw materials employed, and on their behaviour in the course of firing. Examples of such behaviour of a product and of a raw material during high-temperature treatment is demonstrated by figures 6*a-c*. Figures 6*a* and b show the microstructure of flux mixes for the manufacture of wall tiles. One can see the inhomogeneities and gaseous inclusions. The inhomogeneities had their origin in the phonolite employed, showed after firing in figure 6*c*. The size of the bubbles (as measured by the LUCIA system) gives evidence of the course of the firing process. Their size and numbers allow the suitability of a raw material mix for a certain technology to be assessed, as the enclosed bubbles in some products have negative effects on their performance properties (water absorption, strength, etc.).



a)

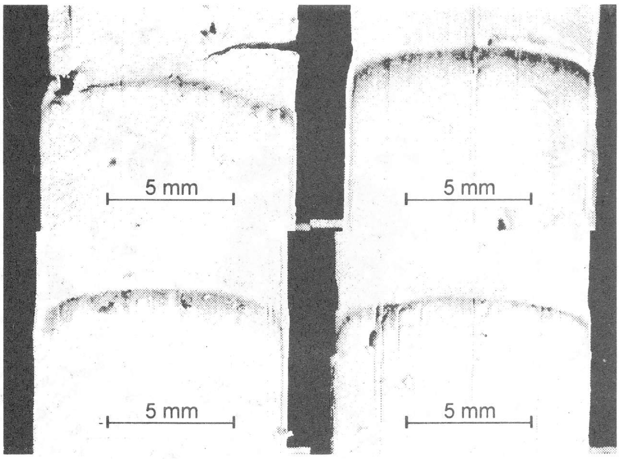
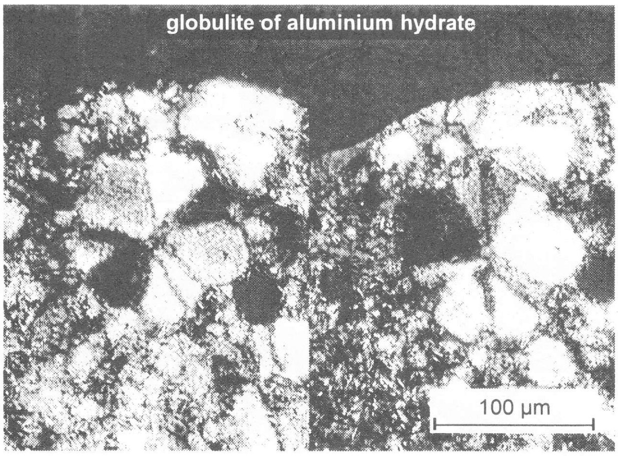


Figure 7. Extrusion velocity profiles in extruded body.



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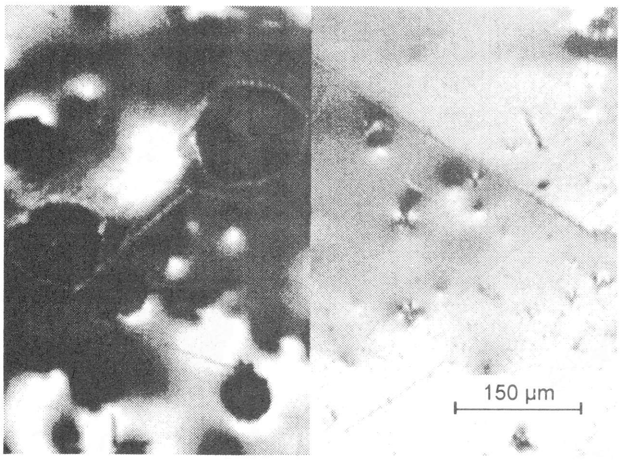
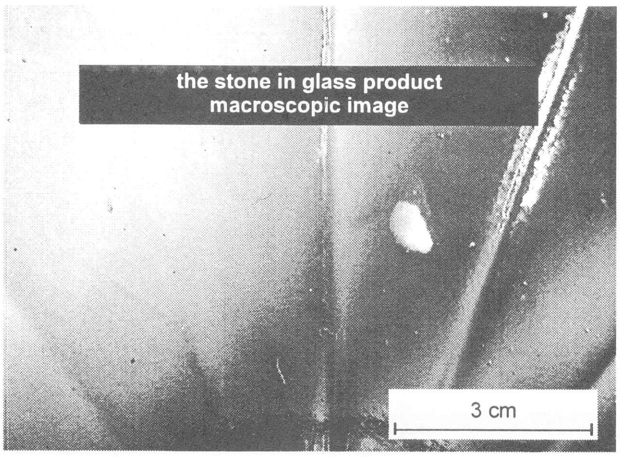


Figure 8. Vitreous glaze on a historical tile.



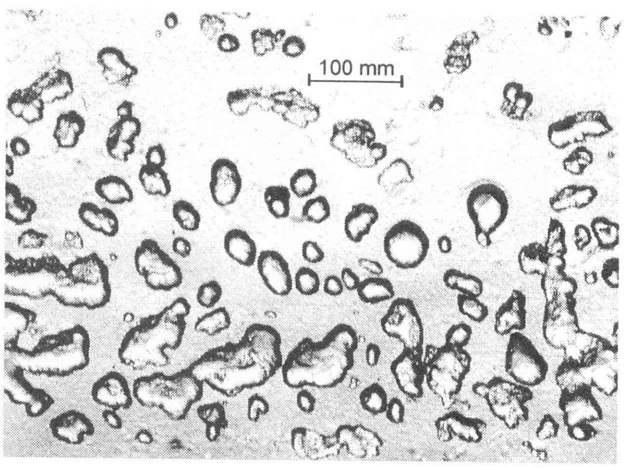
c)

Figure 9.
a) - Grain of electrofused corundum, crossed nicols; *b*) - Globulite of aluminium hydrate; *c*) - Fireclay stone in glass

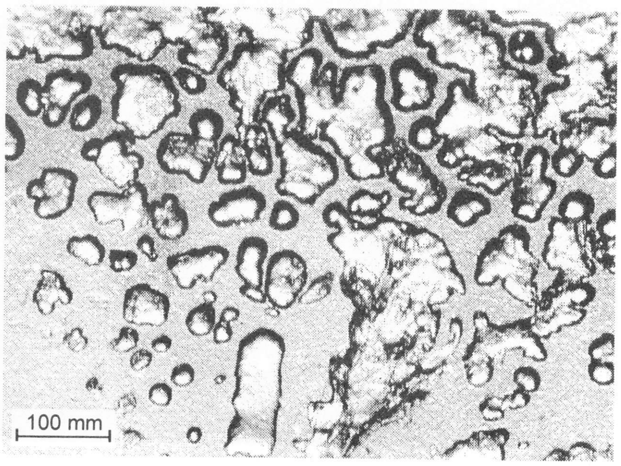
Image analysis can be very useful even in evaluating the forming process. Ceramic products are frequently formed by extrusion. The properties of extruded ware again depend on the microstructure of the surface layers and on that of the body interior. For example, study of the extrusion velocity profiles can be used for evaluating the extrusion process (by measuring and subsequent calculation). The results can then be employed as a basis for modifying the manufacturing technology. Examples of such extrusion velocity profiles are shown in figure 7.

Applications in the field of glass

The LUCIA system provides a number of utilization possibilities also in the field of glassy and glass-ceramic materials. Starting with measurements of optical proper-



a)



b)
Figure 12.
a) - Hydration of a filler in silicone rubber
b) - Hydration of a filler in silicone rubber

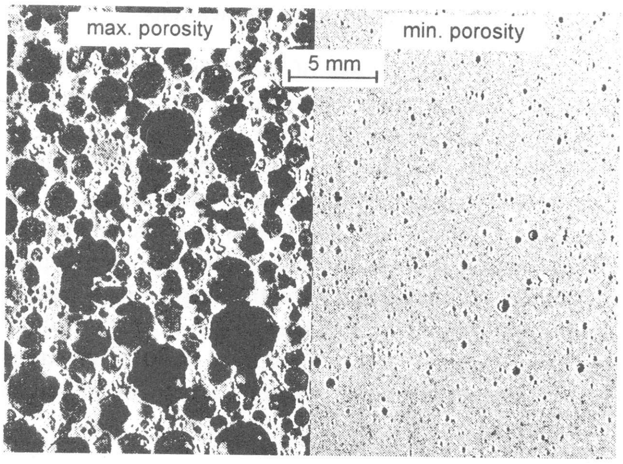


Figure 10. Microstructure of an inorganic foamed material.

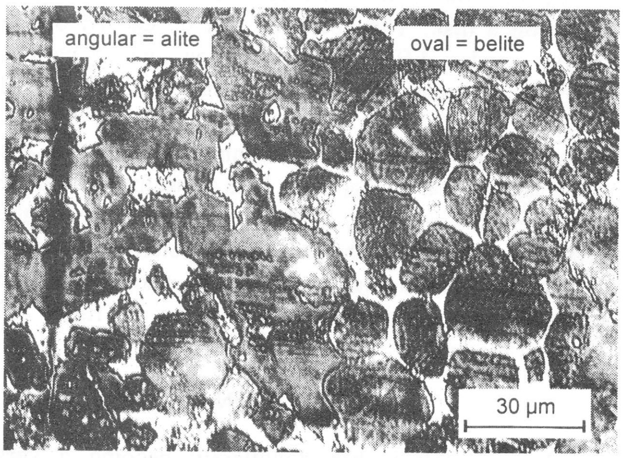


Figure 11. Microstructure of a cement clinker.

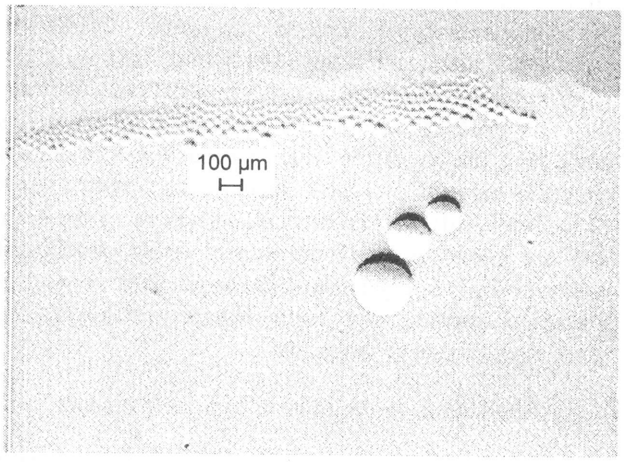


Figure 13. Separation of oil in aqueous suspension.

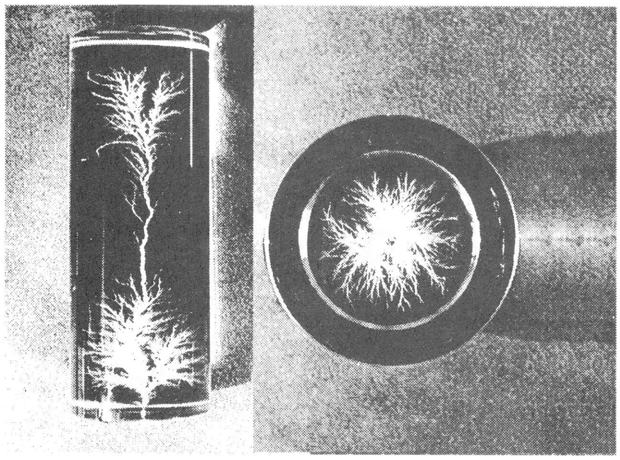


Figure 14. Acrylic glass cylinder after interaction with neutrons (cylinder size 6 cm).

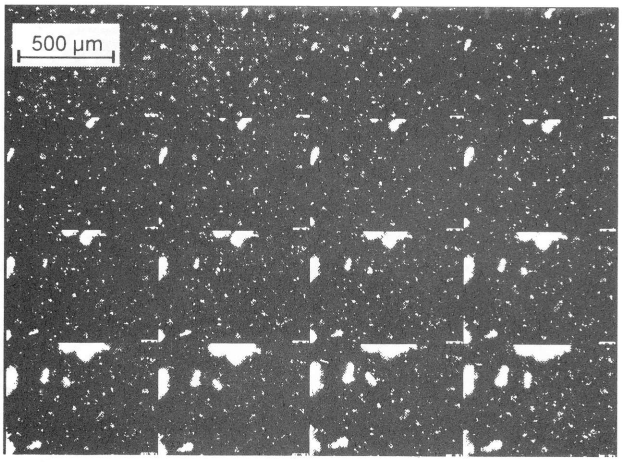


Figure 15. Time sequence of carbonation of $Mg(OH)_2$.

ties and ending with determination of the content and size of defects and crystalline phases (including the archivation). The filed data can be effectively used in the identification of defects in glass thanks to rapid orientation and ready comparing, and in particular in education and all possible kinds of presentations.

Our first example is the evaluation of a vitreous glaze on a tile (figure 8) where various types of defects are easily discernible, such as cracks due to cooling down of the ware (different expansion coefficients of the body and the glaze) and bubbles due to firing. Additional examples include crystalline inhomogeneities (stones) in glasses originating from technological mistakes during glass manufacture (figures. 9*a-c*).

Application in the field of inorganic binders

Numerous applications of image analysis have been found in the field of inorganic binders. Similarly to the cases described above, the properties of materials prepared with the use of inorganic binders depend first of all on their microstructure and phase composition (frost resistance, reactivity, strength, insulating properties, etc.). Examples of such microstructures of foamed inorganic materials are shown in figure 10. An example of an inhomogeneous phase composition of cement clinker (Radotín cement works) is given in figure 11. The actual evaluation is very similar to that used in the case of ceramic materials.

Applications in the field of polymeric materials

Similar applications have likewise been developed for polymeric substances. For example, it is possible to examine hydration of a filler material in silicon rubber, i.e. changes in the particle size of the filler (figures 12*a, b*). The readers would certainly find a number of other suitable utilizations.

Examples of other applications

There is e.g. the case of measuring the size of oil particles during their segregation in an aqueous suspension, which may take place for example in the eye (cf. figure 13). Another example where image analysis can serve for documenting various phenomena, is the creation of archives utilizing further possible techniques of the system, such as shifting, displacing, overlapping and other similar operations with images. Such case is demonstrated by figure 14 where an acrylic glass cylinder had been exposed to the effect of a beam of electrons and subsequently earthed, whereby a network of cracks was formed.

Time-dependent processes

A separate chapter is represented by the study of time-dependent processes. The LUCIA system allows images to be taken at certain preset time intervals (the period of time being displayed in the image) and stored on a disk. The operation can be carried out in various modes differing in resolution and at various intervals of storing the images, to suit best the desired purpose.

At ÚSK, this method of scanning was used in the study carbonation of $Mg(OH)_2$. Lots of useful data can be gained in this way, for example on the course of the process, on changes in the growth of crystalline phases in solutions with changing concentrations, and the like. An example of such a sequence is given in figure 15 (crossed nicols).

CONCLUSION

Image analysis has doubtlessly become a very important tool of materials research and well as of the educational process. As illustrated by the examples, the method is capable of providing very rapidly and effectively a number of useful items of information, allows them to be readily classified and stored, and, in combination with additional software, the results to be retrieved and presented in various forms. For educational purposes, use can be made of illustrative archiving of images including informative texts, as well as of the possibility of simultaneous observation of an object by several viewers (for example in a microscope, and transmission of the visual images to other devices, such as a TV screen, a computer monitor, a projection screen, etc.).

References

1. Amelinckx S., van Dyck D., van Landuyt J., van Tondeloo G.: *Handbook of Microscopy* (Applications in Material Science, Solid-State Physics and Chemistry), Methods II, p. 923-952, Weinheim and New York and Basel and Cambridge and Tokyo 1997.

Translated by K. Němeček.

POUŽITÍ OBRAZOVÉ ANALÝZY LUCIA PŘI STUDIU MATERIÁLŮ

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Součástí materiálového výzkumu je hodnocení mikrostruktury, textury, fázového složení, porozity a dalších parametrů. Příspěvek ukazuje rozmanitost použití analýzy obrazu (AO) v oblasti studia nejen silikátových materiálů.

AO je využívána na ÚSK pro řešení následujících problémů:

keramika
- porozita, obsah jednotlivých fází střepu, zrnitost, orientace částic, interakce s okolím,

sklo
- tavení skla, povrchové interakce, vady skla (vměstky, odskelnění, bubliny),

maltoviny
- obsah jednotlivých fází, tvar a velikost částic, časové změny při reakcích.

Kromě ÚSK využívají AO i další ústavy VŠCHT Praha. Zvláštní kapitolu pak tvoří možnost sledování časově proměnných dějů. Tato aplikace je dokumentována na příkladu sledování karbonatace $Mg(OH)_2$.

AO LUCIA je užitečným pomocníkem jak v materiálovém výzkumu, tak i pedagogickém procesu. Poskytuje velice rychle a efektivně potřebné informace, dovoluje je snadněji třídit a archivovat a umožňuje přehledné prezentace výsledků z nejrůznějších oborů. Pro pedagogické účely je možné využívat nejen ilustrativní archivaci snímků včetně informačních textů, ale i možnost současného pozorování objektu více pozorovateli.

METHODEN DER MINERALOGIE

by Meurig P. Jones
Translated by Manfred Schättle

Ferdinand Enke Verlag Stuttgart 1997, 262 pages, price DM 98.-; ISBN 3-432-27591-9

The book is intended for students and professionals from various fields who use processes of preparation and processing of minerals and rocks and need basic information on the determination of the components of the raw materials. The subject matter does not cover issues useful for experienced mineralogists and crystallographers who can look them up in specialized textbooks. The monograph is consistently devoted to practical utilization of quantitative analytical methods employed in mineralogy and allowing the basic mineralogical data and properties of crystals significant for the industry and various branches of processing to be determined.

The book includes detailed examples of practical exercises which serve for acquiring experience and manual skills, and consequently for gaining confidence in the data established. A special chapter acquaints the reader with mechanical and electronic instruments at present already in general use and indispensable for determining the mineralogical data of diverse types of materials.

Each chapter is divided into several sections. That providing basic information is followed by a brief description of methodical principles and of the instruments employed. The next section presents practical examples and instructions for interpretation of the data measured. Most of the examples are taken from the field of raw materials.

The supplements are devoted to practical exercises and tables of important mineralogical characteristics. A survey of the properties of minerals serves as a basis for a simple scheme for the identification of minerals, suitable in particular for minerals composed of small particles. The given identification methods require no special expert knowledge and make use of simple instruments.

The book is divided into the following ten basic chapters and six supplements: 1 – Introduction into practical mineralogy; 2 – Acquiring of samples; 3 – Classification of minerals; 4 – Identification of minerals; 5 – Polarization microscope in practical mineralogy; 6 – Theory of image analysis; 7 – Modern systems of image analysis; 8 – Interpretation of images of mineralogical samples; 9 – X-radiation, electron rays and other methods of mineralogical analysis; 10 – Purposes of mineralogy and distribution of minerals.
Supplements: 1 – Practical examples and exercises; 2 – Identification of mineral particles; 3 – Series of elements in alphabetical order; 4 – Elements ordered according to atomic numbers; 5 – Selected minerals ordered according to mean atomic numbers; 6 – Minerals ordered according to elements.

The book can be recommended to all establishments dealing with practical mineralogy, processing of raw materials, laboratories in industry as well in schools, and wherever professional standard of knowledge is to be raised and routine operations in mineralogical analyses trained.

M. Maryška