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THE NEUTRON RADIOGRAPHIC THERMAL ANALYSIS (NRTA)

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ABSTRACT

The principle of the new method is based on controlled heating of a sample placed in a neutron beam. The intensity of the neutron beam passing through the sample may be varied due to possible physical or chemical changes caused by heating. A neutronographic image of the heated sample can be continuously observed. For kinetics studies one may take image photographs by proper temperature and time points.

The method is exceedingly convenient for evaluation of processes connected with changes in content of hydrogen compounds, especially of water. The NRTA has been applied to a topographic analysis of bounded hydrogen distribution changes throughout a cylindriform block of kaolinite.

INTRODUCTION

The neutron radiography as a non-destructive method for visual testing of solids can yield important information not obtainable by more traditional methods /1/. The neutron radiographic analysis, similarly to the X-ray radiography, provides an internal image of an object. Neutrons passing through this object are absorbed and scattered with a different intensity in various parts of the specimen cross section. The obtained image is a topography of a geometrical distribution of material layers with certain mass neutron beam attenuation coefficients. Elements with nuclei showing the highest attenuating effect on a beam of neutrons are gadolinium, boron, hydrogen, lithium, cadmium, samarium and europium.

The proposed neutron radiographic thermal analysis (NRTA) multiplies the mentioned advantageous possibility by extending the neutron radiography on studies of processes dependent on both time and temperature. First of all, the NRTA has been developed as a successive series of neutron radiographic analysis of an object in which, due to the controlled heating, structural and chemical changes occur on the condition that these changes are closely connected with changes of mass attenuation coefficient for neutrons. An objective example of such processes may be water concentration and distribution changes in a mineral building material on almost each step of its fabrication.

The neutron radiography thermal analysis allows to gain interesting images of relatively large-sized samples or objects.

THE PRINCIPLE OF THE NEUTRON RADIOGRAPHIC THERMAL ANALYSIS

A collimated neutron beam from a proper neutron source is directed onto the object to be radiographed. This object is placed in a furnace, and its tempe-

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rature is controlled by means of a programmable regulator. Heating may cause some changes in structure and chemical compositions of the studied sample. If no changes take place, the object will modify the neutron beam by scattering or absorbing the radiation, and the beam reaching a detector will have an intensity pattern representative of the structure and composition of the object. Provided that the internal changes of the object caused by heating exert an influence on the values of mass attenuation coefficients, these changes can be made perspicuous comparing successive series of neutron radiographs taken during heating.

Although the neutron radiographic method is similar to the X-ray radiography, it is not possible to use the direct photographic technique common for X-ray radiography, neither for detection and recording of neutron radiographic image, owing to a very little direct effect of neutrons on photographic film. This is the reason why an intermediate foil which converts the neutron image into strong ionising radiation is used, and it is this secondary emission which is detected by a photographic film. Similarly, a television system for neutron radiography needs a scintillator screen behind studied object, as well as an image intensifier placed between the screen and a TV camera. The neutrons are converted into the light signals. These being intensified may be seen on a TV monitor. Instead of the scintillator a screen made of gadolinium may be used for the front window of an intesifier tube. In this case the neutrons are converted into secondary electrons which being accelerated form an image on a scintillator screen. The TV technique provides proper devices for studies of dynamic systems and processes.

NRTA STUDY OF THE KAOLINITE CONVERSION TO METAKAOLINITE

The dehydroxylation of kaolinite and formation of metakaolinite according to the reaction

$$Al_4(OH)_8Si_4O_{10} = 2(Al_2O_3 \cdot 2SiO_2) + 4H_2O$$

occurs between 450 and 600 °C. Due to this reaction several simultaneous processes, such as formation and diffusion of water, volume changes of solid products and water vapour release take place. As a result the concentration of hydrogen atoms in solid kaolinite decreases with progress of these processes.

As hydrogen is a high neutron-attenuation cross-section material its escape in the form of water vapour can be observed by means of a device for NRTA.

A cylindriform block of a dried china clay (kaolinite) of 40 mm in both diameter and length was prepared and placed in a furnace, so that the collimated neutron beam from the nuclear reactor VVR-S could pass through it in the axial direction.

The block was subjected to heating program of 1.5 $K.min^{-1}$ and the temperature was successively maintained at 400, 500 and 600 °C for one hour a step. The neutron radiographs were taken at the end of each step. The exposure time was 15 minutes. Both the radiographs and appropriate densitogrammes of the images are shown in Fig. 1. One may easily distinguish the sample of kaolinite from that formed by metakaolinite only. The middle radiograph showes a hydrogen atoms radial concentration gradient in the internal vicinity of the outer surface of the cylindriform block.

CONCLUSION

The described NRTA seems to be an useful tool for studies solid materials containing hydrogen in any chemical form at elevated temperatures. Particularly, it concerns cases whan a change of hydrogen content caused by heating takes place. The method offers its utilisation not only in the mentioned field of silicates technology, but also for study of other important materials, e.g. organic polymers, fuels and non-silicate ceramics.

REFERENCE

1 Neutron Radiography Handbook, Eds.: P. von der Hardt and H. Röttger, D. Reidel Publishing Comp., Dosdrecht-Boston-London, 1981.

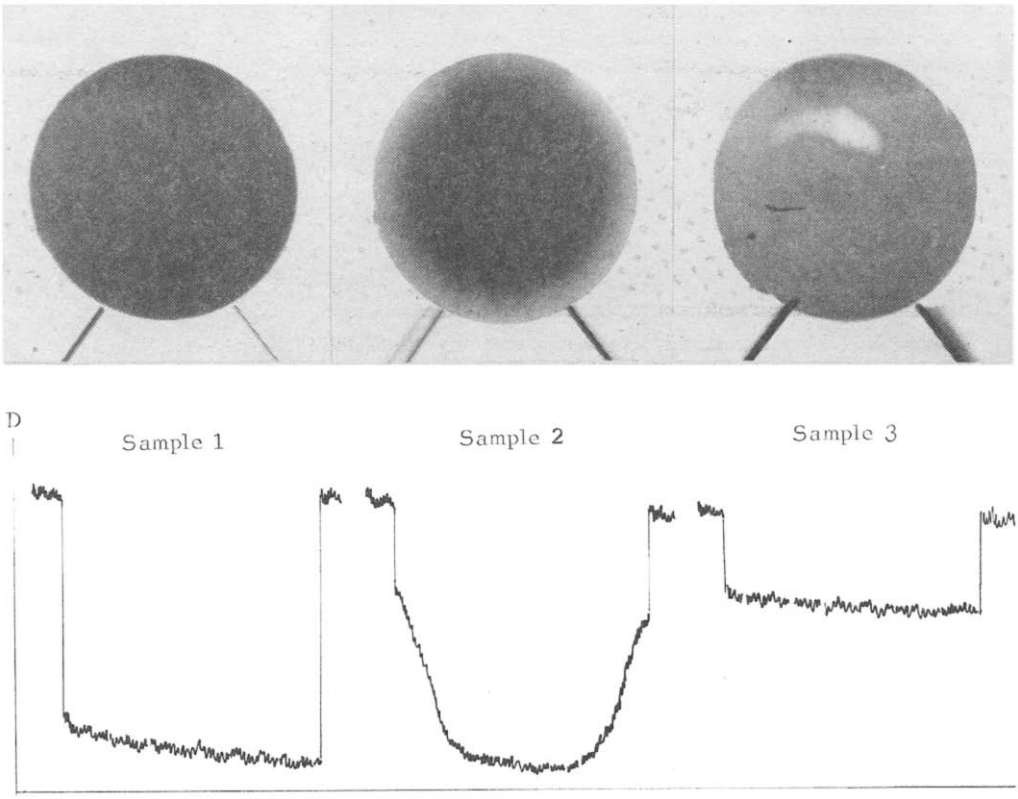

Fig. 1 Photographic density D distribution of neutronographs of ceramic samples