



WORLDWIDE OLDEST PRIVATE LABORATORY SPECIALIZED FOR  
GENUINE ANALYSES

FH Koblenz    FB Werkstofftechnik Glas und Keramik.



## **Remodeled    ?**

### **manufactured from old pulverized clay terra cotta?**

Bonding agent - resins suspected/detected in terra cotta, what does this mean?

Varying clay, varying clay colours, what does this tell us about the object made of fired clay?

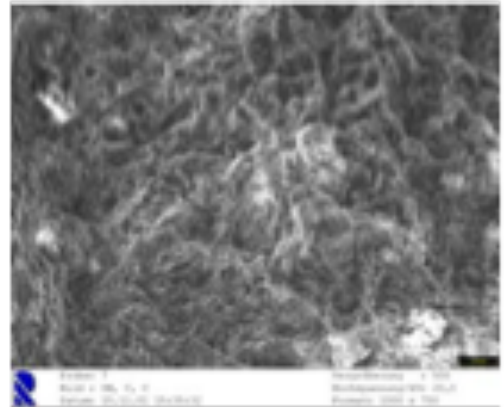
Belonging of varying parts e.g. head to body, have original parts being joined, or not ?

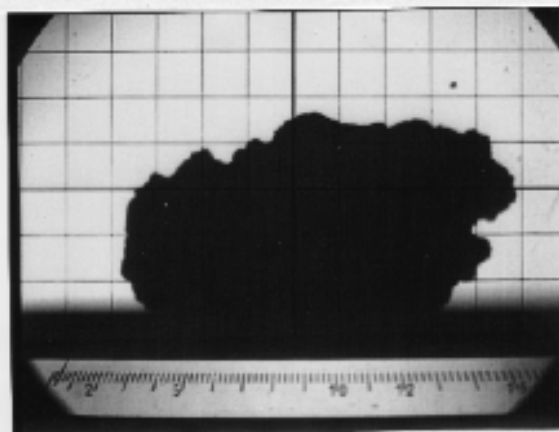
These questions which have been occupying and influencing the Asian and African art market intensively, require urgent clarification and remedying. The thermoluminescence test "T L" can detect the age of fired clay, for the other points and questions, scientific, neutral analysis procedures are of assistance.

I will be issuing shortly an extensive article which I have prepared together with Prof. Dr. Klein of the University of Koblenz "Höhr-Grenzhausen"- faculty "Ceramic and Glass" on this subject and the entire complex of questions mentioned above and below.

To merely claim "Varying clay and varying colours were determined", does not prove from a ceramic/technical point of view that an object must have been manufactured from varying parts.

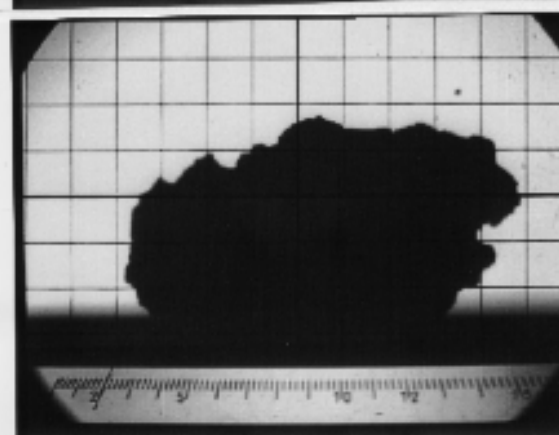
The fact that parts of an object "belong" to the entire object can largely be clarified by the procedure which I will now briefly describe.



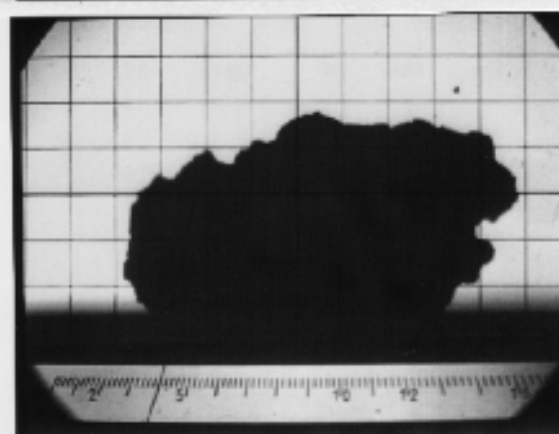


Anlage 5

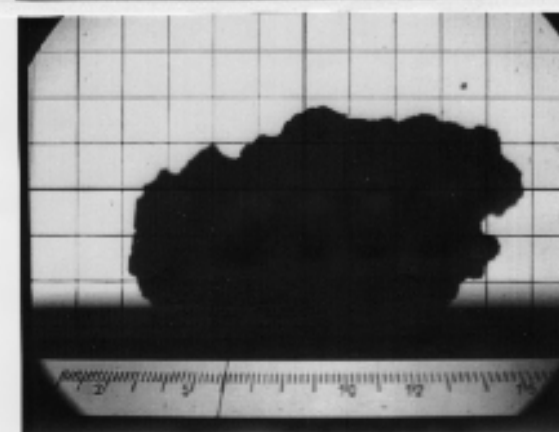
20 °C



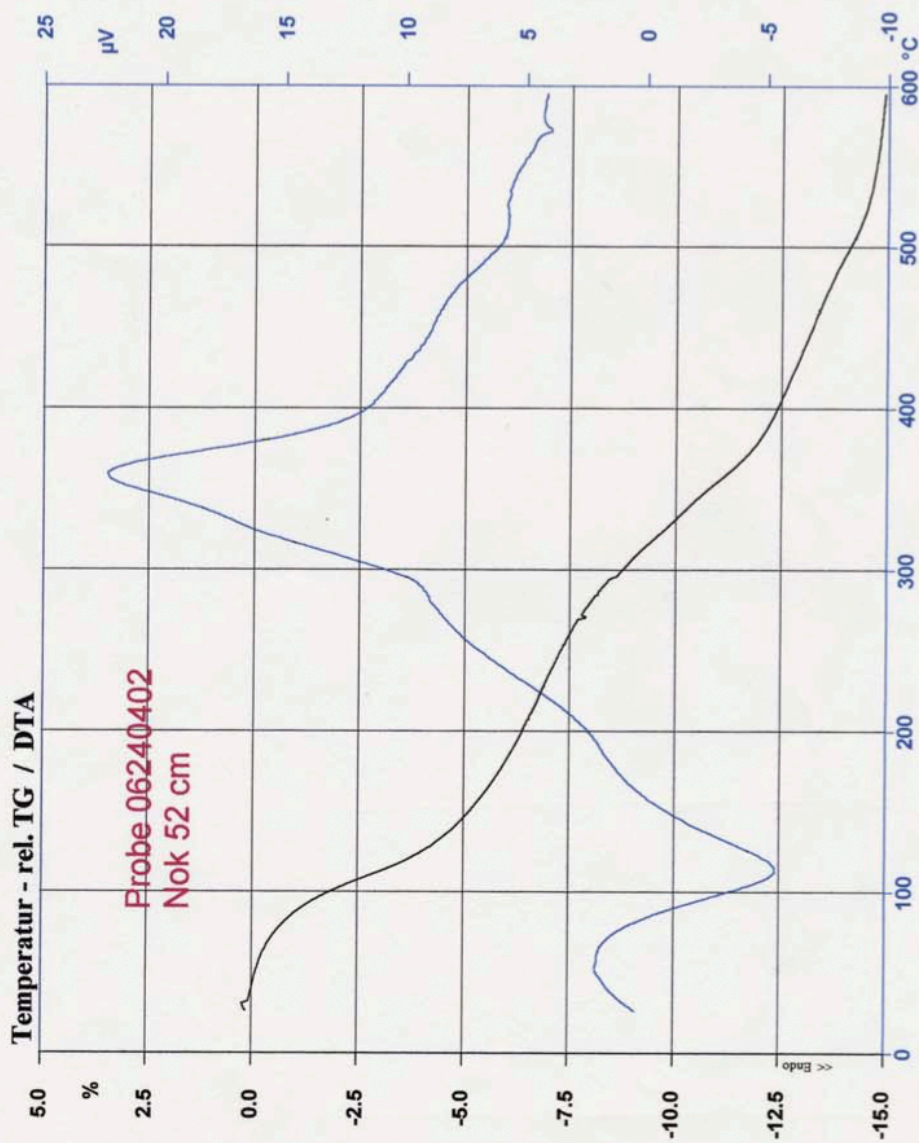
200 °C



400 °C



600 °C



Gerät : STA502  
Versuchsname : 06240402  
Datum : 07.05.02  
Probenmaterial : nok 52  
Probengewicht : 134 mg  
Referenzmaterial : Al2O3  
Referenzgewicht : 253 mg  
Meßsystem : AL203  
Atmosphäre : luft  
Max. Temperatur : 601.3 °C  
Temperaturprogramm : 600-10  
Korrektur DTA : 0000juli600  
Korrektur TG : 0000juli600  
Benutzer : mli





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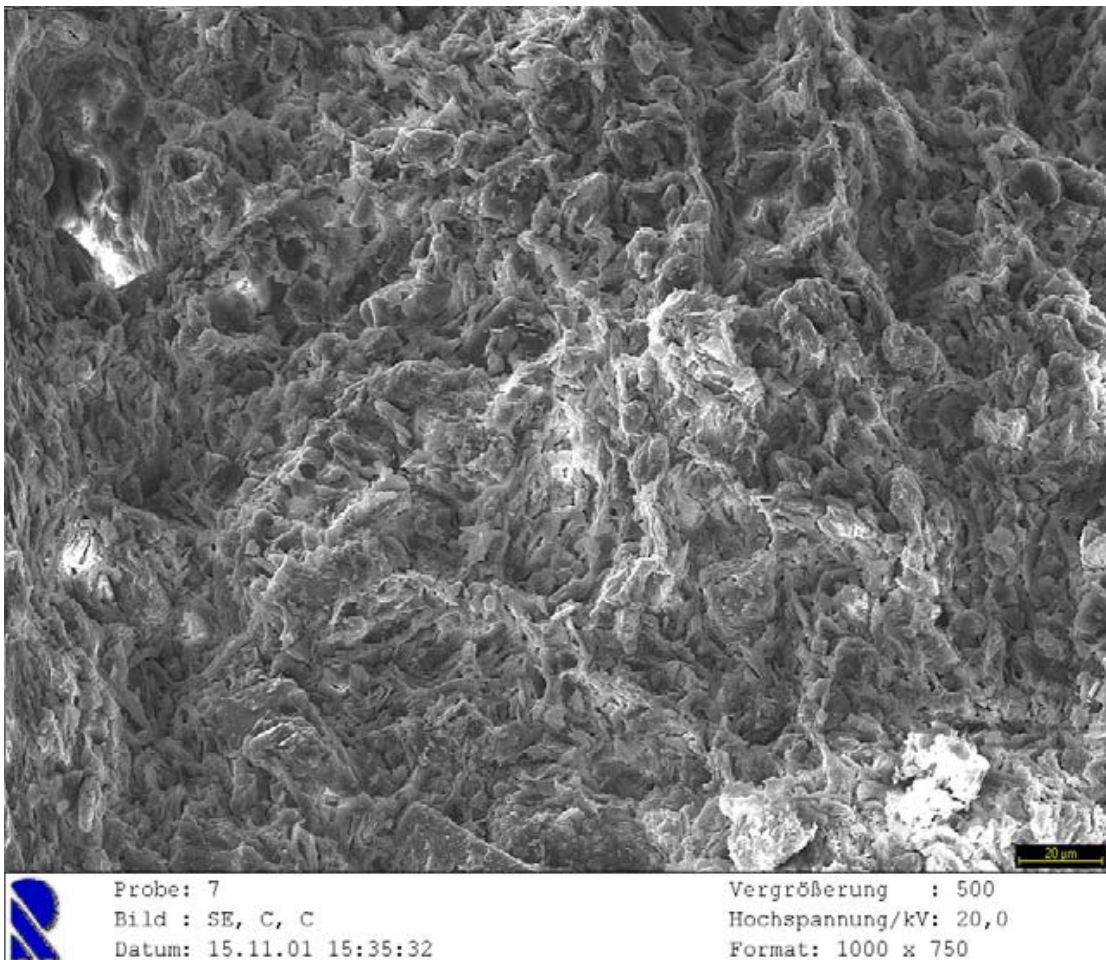
Hochspannung:

Probe: Kotalla Test rot 20.0 kV

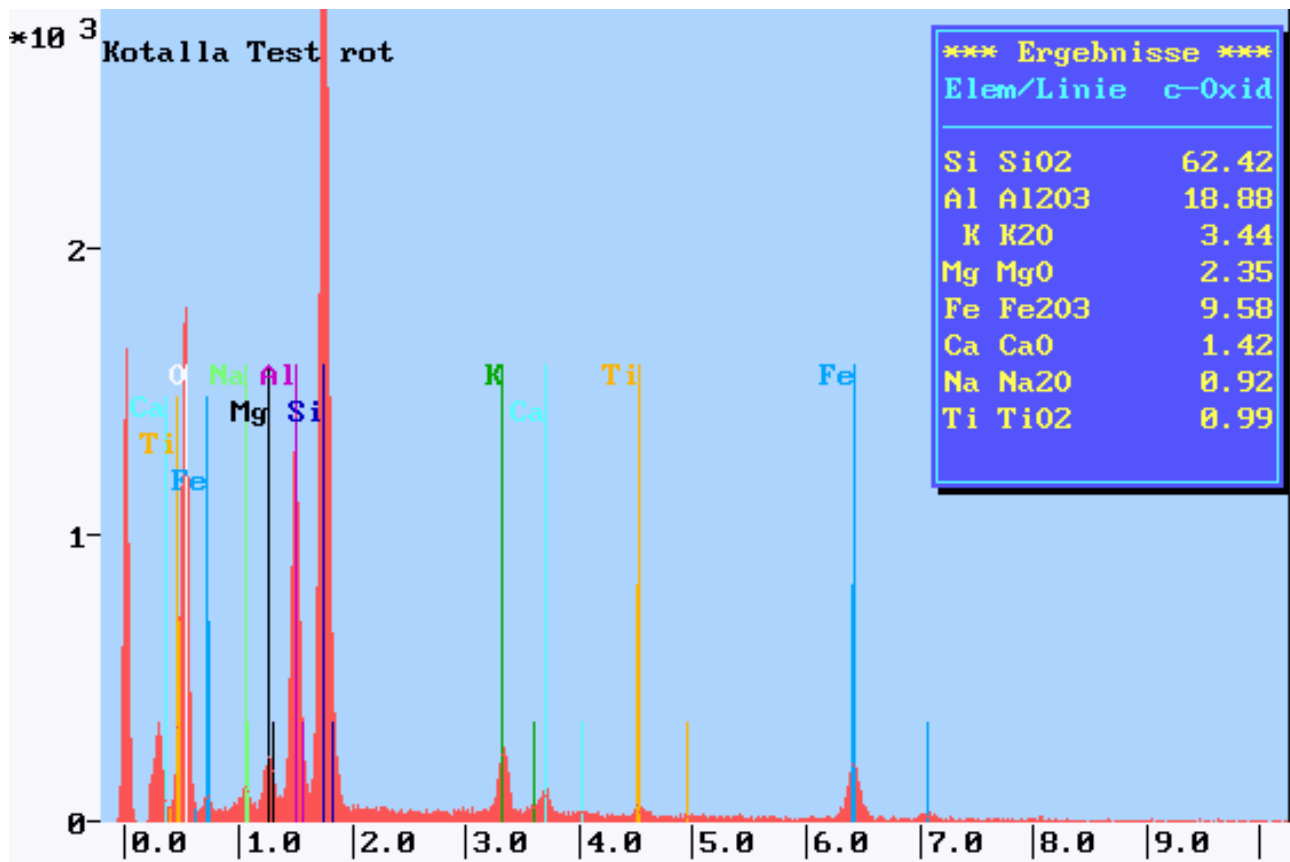
Datum: 15.11.2001 Bearbeiter: Dipl.-Ing.(FH) A.Gros

Elektronenbild:

1



## Spektrum:



## List of applied examination methods

### 1. Light-optical microscopy

Measuring instrument: Measuring method: Magnifications:

Photo: Stereo microscope Wild M 8

Reflected light microscopy

13.5 x and 37.5 x

High gloss photo from Kodak film material Gold 100

## **2. Scanning electron microscopy (SEM)**

Measuring instrument: Measuring method:

Magnification: Print out:

Scanning electron microscope JEOL T 330 A

- Sputtering of specimen with carbon in vacuum - Bonding specimen to object carrier

- Inserting specimen into SEM

- Structural examination with an acceleration voltage of electron-beam of 20 keV

150x and 500x

Micrograph with ink jet printer

## **3. Chemical analysis with energy dispersive X-ray analysis (EDX)**

Measuring instrument: Measuring method:

EDX analyser RÖNTEC

Analysis with energy-dispersive X-ray and recording of X-ray spectrum

EDX spectrum and quantitative, standard-free chemical analysis with ink jet printer

Print out:

## **4. Thermal gravimetric analysis (TG) and differential thermal analysis (DTA)**

Measuring instrument: Measuring method:

Print out:

5. Heating microscopy Measuring instrument:

Measuring method:

Print out:

BÄHR STA 502 (simultaneous thermal analysis)

The specimen is thermally treated reduced in size ( < 100 µm) from ambient temperature to 600 /C at a heating speed of 10 K/min. Mass loss during heating is determined by TG and the endothermic and/or exothermic reactions by DTA. Organic substances in ceramic structure can be detected. Diagram with mass loss and reactions as function of temperature (ink jet printer) Leitz heating microscope with SONY video recording and video graphic printer.

The core drill specimen is thermally treated in the heating microscope from ambient temperature to 600 /C on an aluminium oxide base at a heating speed of 10 K/min. The changes to the specimen are registered with increasing temperature.

Video print of specimen in initial state and after 100 to 600 /C in 100 K stages.

The claim or proof that resin bonding agents are in the structure of terra cotta, do not prove that an object was manufactured from old pulverised terra cotta. Reliable proof can be provided with the following method of examination in the course of an overall test report:

Structural analysis using a light microscopy and scanning electron microscopy REM, chemical analysis with energy-dispersive X-ray analysis, thermal gravimetric analysis and differential thermal analysis - TG + DTA and heating microscopy.

A list of the procedures and representations of measurements are enclosed in the annex.

The following statements can be made:

This is a fired ceramic structure = terra cotta

This is not a fired structure of pulverised substances!

This is a ceramic structure with portions of bonding stabilising agents !

As far as C is concerned, this may be a case of supplementary measures which are absolutely necessary, e.g. to prevent terra cotta which was fired at too low a heat from disintegrating (risk of ground/air transport) or it is a conservation measure to maintain/ supplement the surface.

Should you have any questions, suggestions or wish to exchange views, obtain further information or are just interested in the publication which I will be issuing in Cupertino with the University of Koblenz, please do not hesitate to contact me.

Kind regards, Your

Ralf Kotalla

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