Determination of Microhardness and Elastic Modulus of Coal Components by Using Indentation Method

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ABSTRACT: The Vickers microhardness of vitrinite VH is the standard physical property of coal. Micro Hardness Tester CSM Instruments enables the measurement of microhardness continually in the rank from 10 mN to 30 N and particularly the determination the elastic modulus of vitrinite or other coal macerals. This measurement makes possible to determine the critical indentational load for crack initiation of coal with different coalification. Length and orientation of cracks are analysed by image analysis.
KEY WORDS: coal, microhardness.

Introduction

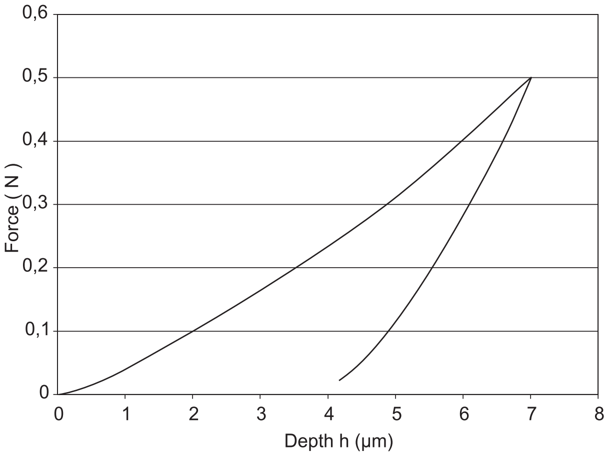
The microhardness is the standard testing parameter of metals (Kašpar 1988), ceramics (Dietz and Tietz 1990) and composites. Microhardness testing is useful for testing very thin materials like foils and measuring individual microstructures within a larger matrix. This parameter is applied for testing of coal, too (Eremin et al. 1980, van Krevelen 1993).

The microhadness of coal from the Upper Silesian Coal basin was studied in past by various authors (Beneš 1957, Beneš and Salava 1960, Das 1968). Martinec and Kožušníková (2006) processed the older data, but in the last time more detailed analysis has not been done. The Institute of Geonics of ASCR got Micro Hardness Tester CSM Instruments last year and since the research of coal microhardness and elastic modulus has been started.

Method of measurement

The equipment Micro Hardness Tester CSM Instruments (Fig. 1) enables in addition the measurement of standard microhardness to determine even elastic modulus of tested material. Typical course of measurement curve of vitrinite is shown in the Fig. 2.

■ **Fig. 1.** Micro Hardness Tester CSM Instruments with image analysis processing LUCIA DI.



■ **Fig. 2.** Typical graph of dependence of indent depth on indentor force F_{IT}.

It is possible to calculate the elastic modulus from unloading curve by two ways:

- Tangent Method
- Power Law Method (Oliver and Pharr)

The Tangent Method is also called the Linear Extrapolation Method (Doerner and Nix 1986). This assumes that the first portion of the unloading curve is linear and simply extrapolates linear portion to intercept the displacement axis. This method is applicable to materials that show a high degree of stiffness and a large deformation so that the unloading curve is, to a good approximation, linear. The Power Law Method recognizes the fact that the first portion of the unloading curve may not be linear, and can be described by a power law relationship (Oliver and Pharr 1992). The Power Law Method was used for calculation of elastic modulus E_{IT} of tested coal samples:

$$E_{IT} = \frac{1-\nu_s^2}{\frac{1}{E_r} - \frac{1-\nu_i^2}{E_i}}$$

E_i = elastic modulus of the indenter (1141 GPa)
ν_i = Poisson' ratio of the indenter (0,07)
E_r = reduced modulus of the indentation contact
ν_i = Poisson' ratio of the sample (user's selection)

The test samples should have a smooth surface and be fixed perpendicular to the indenter. Experiments were realized on polished sections of coal piece with various degree of coalification.

The load was applied by a force–controller at a rate of approach speed 30 %.min^{-1} until the maximum load was reached. The maximum loads of 0.1, 0.25, 0.5, 1, 1.25, 1.5, 2, 3 and 5 N were used. Five indents were made at maximum load conditions on each sample.

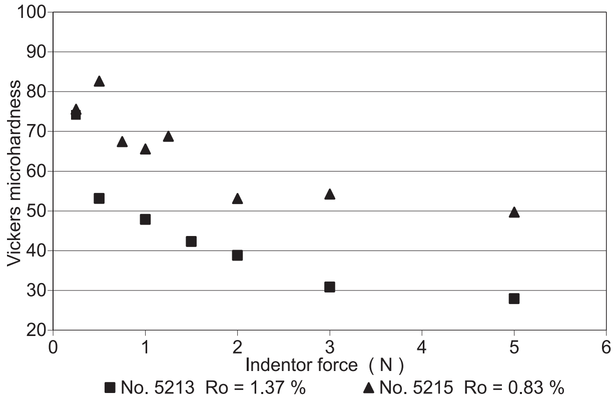
Immediately after loading the indentation impressions were examined with an optical microscope and the images of impression were taken to minimize the effect of the environment on crack growth. The optical part of equipment is interconnected with image analysis processing LUCIA DI. This software enables to measure crack length and orientation to determine of critical indentation load for crack initiation.

Details of sample localities, maceral composition and vitrinite reflectance (R_0) are specified in the Table 1.

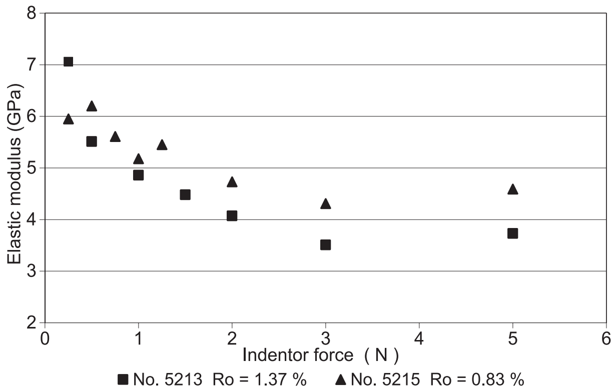
Results and discussion

Obtained results are presented in graphs (Figs. 3, 4, 5) displaying the change of microhardness HV, elastic modulus E_{IT} and crack length for various maximum indentor force F_{IT}.

It is evident that both the Vickers microhardness and elastic modulus decrease with increasing indentor force. The corresponding values of Vickers microhardness and elastic modulus



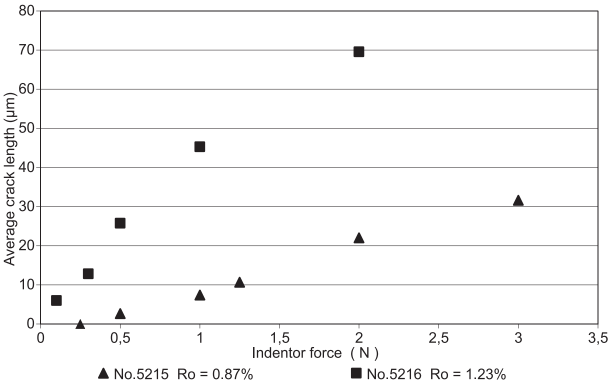
■ **Fig. 3.** Dependence of Vickers microhardness HV on indentor force F_{IT} for coal samples with different reflectance of vitrinite R_0.



■ **Fig. 4.** Dependence of elastic modulus E_{IT} on indentor force F_{IT} for coal samples with different reflectance of vitrinite R_0.

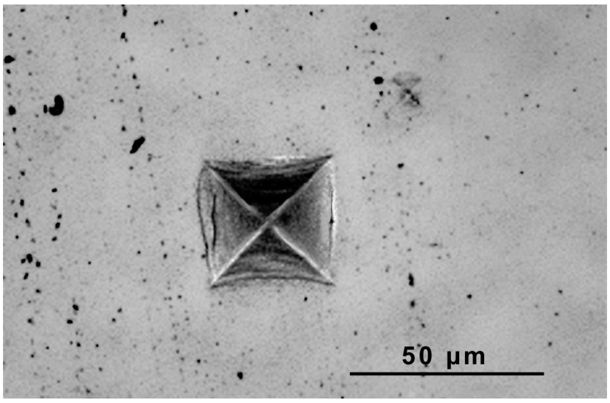
Sample No.	Mine	Borehole No.	Stratigraphic horizon	Maceral composition [%]			R_0 [%]
				Vitrinite	Liptinite	Inertinite	
5213	Staříč	1111	Hrušov Mem.	79.8	0	20.2	1.37
5215	Lazy	C 110	Saddle Mem.	69.0	7.9	23.1	0.83
5216	František	F3313	Poruba Mem.	87.6	3.8	8.6	1.23

■ **Tab. 1.** Localisation, maceral composition, vitrinite reflectance of tested samples.

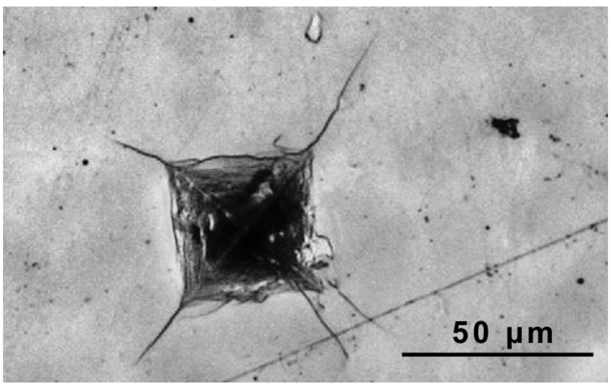


■ **Fig. 5.** Dependence of average Palmquist crack length on indentor force F_{IT} for coal samples with different reflectance of vitrinite R_0.

for coal sample of R_0 = 1.37 % are lower than for coal sample of R_0 = 0.87 %. The average Palmquist crack length is also different for coal samples with different degree of coalification. The Palmquist crack length on coal of R_0 = 1.23 % is considerably longer than on coal of R_0 = 0.87 % (Fig. 5). The critical indentational load for crack initiation is possible to determine in the Fig. 5, too (sample 5215 – 0.5 N; sample 5216 – 0.1 N). In Figs. 6 and 7 there are shown the indents on coal samples with various degree of coalification. The indent at indentor force 0.5 N on coal with lower coalification has not Palmquist cracks unlike the indent on coal with higher coalification at the same indentor force.



■ **Fig. 6.** Indent on maceral of vitrinite group, sample No. 5215, F_{IT} = 0.5 N, R_0 = 0.87 %.



■ **Fig. 7.** Indent on maceral of vitrinite group, sample No. 5216, F_{IT} = 0.5 N, R_0 = 1.23 %.

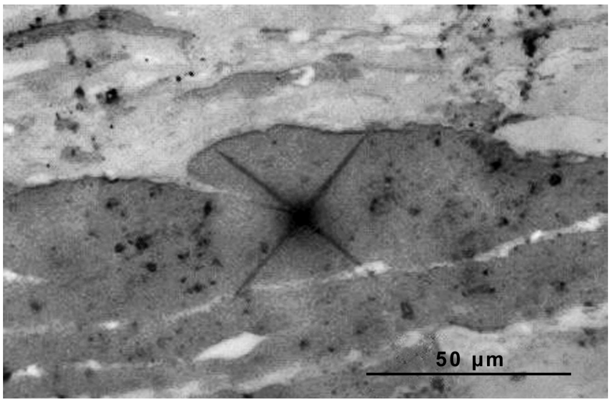
Maceral group	E_{IT} [GPa]	HV_{50}	Character of Indent
vitrinite	6.20	82.68	Pyramid
inertinite	7.11	94.40	Cross
liptinite	4.43	55.66	Cross

Tab. 2. Microhardness HV, elastic modulus E_{IT}, character of indent – sample 5215.

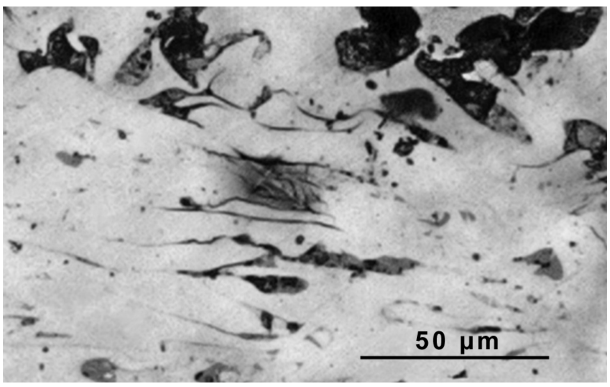
The values of microhardness HV and elastic modulus E_{IT} of further components of coal – macerals of inertinite and liptinite group for the same sample are different (Table 2). The microhardness and elastic modulus of the macerals of liptinite group is lower than macerals of vitrinite group, the microhardness and elastic modulus of macerals of inertinite group is higher than macerals of vitrinite group. Mainly the character of indent is different – see Figs. 8 and 9. The indents on megaspore show the considerable elastic deformation and the indents on macerals of inertinite group are very often anisotropic.

Conclusion

The indentation elastic modulus E_{IT} and Vickers microhardness at various indentation load of vitrinite with different degree of coalification from the Upper Silesian Coal basin have been determined. Vickers microhardness and elastic modulus decrease



■ **Fig. 8.** Indent on maceral of liptinite group, sample No. 5215, F_{IT} = 0.5 N.



■ **Fig. 9.** Indent on maceral of inertinite group, sample No. 5215, F_{IT} = 0.5 N.

with increasing indentor force. The corresponding values of Vickers microhardness and elastic modulus of coal sample with higher coalification (R_0 = 1.37 %) are lower than of coal sample with lower coalification (R_0 = 0.87 %).

The Palmquist crack lengths have been measured and the critical indentation load for crack initiation has been determined. The critical indentation load for coal sample with higher coalification (R_0 = 1,23 %) are considerably lower than for coal sample with lower coalification (R_0 = 0.87 %).

Acknowledgement

The presented work was supported financially by the Academy of Sciences of the Czech Republic by the Research plan No. AVOZ 30860518.

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