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ANOMALOUS MICROHARDNESS IMPRESSIONS OF SOME WEATHERED COALS

by

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In a recent publication[1] it was reported that microhardness impression on different North American coals (Carboniferous age) by the Vickers Hardness Test could possibly be used to detect oxidation which transformed the plastic state of fresh vitrinite to an elastic state. By plastic state we mean that the impression of the Vickers hardness prism remains after the indenter is withdrawn, whereas in the elastic state the impression largely disappears leaving two crossed lines due to the fact that the indentation has rebounded into the horizontal plane. This transformation from the plastic to the elastic state occurred rapidly in high volatile coals, but more severe oxidation conditions were required to cause this change in low volatile coals. Reflectance also increased with oxidation.

Alpern[2] mentioned that he did not observe any change in microhardness impressions of an 18-year old weathered (oxidized) French coal of Stephanian age following the method described by Nandi et al[1]. The microhardness impressions were the same in all locations even at the edge of the particles exposed to air. The lack of variation in the impressions suggested that the transformation from plastic to elastic state was ineffective in detecting the oxidation of this particular coal.

The object of the present study was to explore further the ability of the microhardness impression test to detect the oxidation of coals of different geological age and rank and also to determine the cause of the failure of the vitrinite of certain coals to become elastic after oxidation.

The development of elastic properties in the vitrinite was interpreted to be due to a chemical effect, namely the formation of oxygen cross links in the Harbour Seam (Nova Scotia coal) vitrinite studied previously[1]. The failure of vitrinite to develop elastic properties on oxidation appeared to be associated with certain structural features, that is to say, a telinite structure and the presence of bitumen and fluorescent material in the vitrinite.

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One French coal from the upper Stephanian age and two Indian coals from the Gondowana age were selected for this investigation. The Aumance coal from France is a special coal, bordering between sub-bituminous coal and lignite, which was weathered for 18 years[3]. The two coals from India are bituminous, one from east Bokaro, Bihar and the other from Ranigunj, West Bengal. These were also weathered for more than 12 years.
The physical, chemical and petrological analyses are given in Table 1.

Methods and Procedure

Induced oxidation of the coal sample (particle size 0.6 to 0.8 mm) was carried out in an air circulated oven at 105°C. The coal particles were then mounted and polished for testing the plastic and elastic properties. More severe oxidation was carried out by heating the particles in air under an infra-red lamp for a specific time according to the method described previously[4].

Discussion

Aumance Coal

Negative dilatation, high contraction and the high plasticity index indicated that this coal was partially oxidized. A considerable amount of telinitic structure and fine micrinite were observed during microscopic examination as shown in Figures 1 and 2, respectively. The diagonal length of the microhardness impression in the structureless vitrinite (A in Figure 3) of this weathered coal appears to be slightly smaller than in the structured vitrinite (B in Figure 1). After 48 hours of induced air oxidation at 105°C, the change in the microhardness impression, on progressing from the centre to the outside edge of the particles of structured vitrinite (Figure 4) shows no significant change. On severe oxidation under an infrared lamp for 1.5 hours, only very slight change in the microhardness impression on the structured vitrinite on progressing the centre to the edge was noticed, which means essentially no transformation from the plastic state to the elastic state occurred (Figure 5). But in another part of the same sample the impression on the structureless vitrinite at the edge in Figure 6 appears to be indistinct (elastic), compared with the deep impression at the centre B (plastic). The progressive transition from the shallow impression at the edge to the distinct deep impression at the centre suggests formation of an elastic state from the plastic state due to oxygen penetration. This type of transition from edge to centre is almost absent in weathered vitrinite with tellinite structure and associated fine micrinite. It was noted in this coal that a considerable amount of exinite, resinite and fluorescing vitrinite were observed under a fluorescent light microscope[7].

Bokaro Coal

This coal was obtained from the Kargali Seam. Nil dilatation, slight contraction and a Free Swelling Index of 1 of this sample indicate that this coal is extensively oxidized. The fresh sample is known to possess excellent coking properties[5]. Microscopic investigations revealed that the structure of vitrinite is similar to that of telinite (Figure 7). The dendritic material (dark material) in Figure 7 appears to originate from resinitic or bitumen-like materials. This is confirmed by the slight fluorescence observed under the fluorescence light microscope[7].

The unchanged microhardness impressions from the edge to the centre of the original weathered coal and the induced 48-hour air oxidation of this coal are very similar to those of Aumance coal (Figure 4). The plastic impressions are observed in all places on structured vitrinite even after induced oxidation for 48 hours. Again it was noted that there was resinitic or bitumen-like material in the vitrinite. Reflectance values are also irregular from the edge to the centre.

Ranigunj Coal

This weathered coal was acquired from the Poniati Seam. Nil dilatation, slight contraction and a Free Swelling Index of 1 suggest that the coal was converted to non-coking coal because of oxidation, although the freshly mined coal possessed excellent coking properties[5]. The vitrinites are structureless and homogeneous. The microhardness impression is very shallow and indistinct at the edge and the same type of impression continues progressively from the edge towards the centre (Figure 8). The elastic impression in all the vitrinite particles suggests that this coal is highly oxidized. A variation of average reflectance from 0.53% to 0.73% was observed in this coal but no systematic increase of reflectance from the centre to the edge, as described in the previous paper[1] was noticed.

At this stage in the investigation it was evident that the transition from the plastic to the elastic state of vitrinite is not a reliable indication of the state of oxidation of vitrinite. Vitrinite in different coals behave differently on weathering in this regard. There is some indication that those vitrinites which remain elastic after weathering possess a telinite structure and there is a considerable amount of imbedded bitumen-like or resinous materials in the vitrinite. The authors would like to pursue this matter further and would welcome the receipt of fresh samples of 250 gm coarse coal (more than 2" in particle size) for further study.

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TABLE 1

Proximate Analysis

		Aumance Coal	Bokaro Coal Kargali Seam	Ranigunj Coal Poniati Seam
Moisture	wt %	3.5	1.7	3.6
Ash	wt %	18.4	3.2	9.8
Volatile Matter	wt %	32.6	33.8	38.9
Fixed Carbon	wt %	45.5	61.3	47.7
		100.0	100.0	100.0
Free Swelling Index		1	1	1
Carbon		80.2	82.9	83.1
Hydrogen		5.5	5.9	5.6
Dilatation Test				
Softening Temperature,	^{o}C θ_S	362	nil	nil
Contraction,	% C	35	2	1
Max Temperature of Contraction,	^{o}C θ_S	431	nil	nil
Dilatation,	^{o}C	-31	nil	nil
Max Temperature of Dilatation,	^{o}C	447	nil	nil
Plasticity Index, $\frac{C}{\theta_C-\theta_S}$		0.51	nil	nil
Petrographic Analysis				
Vitrinite	wt %	50.2*	56.6	64.9
Exinite	wt %	22.6†	4.9	5.1
Semi-fusinite	wt %	9.8	33.0	25.4
Fusinite	wt %	13.6	3.2	1.9
Micrinite	wt %	3.8	2.3	2.7
		100.0	100.0	100.0

* Tellinite - 9.4 † Resinite - 20.8

LIST OF FIGURES

FIGURE 1 - Aumance Coal
Telinite Structure, air (x 500)

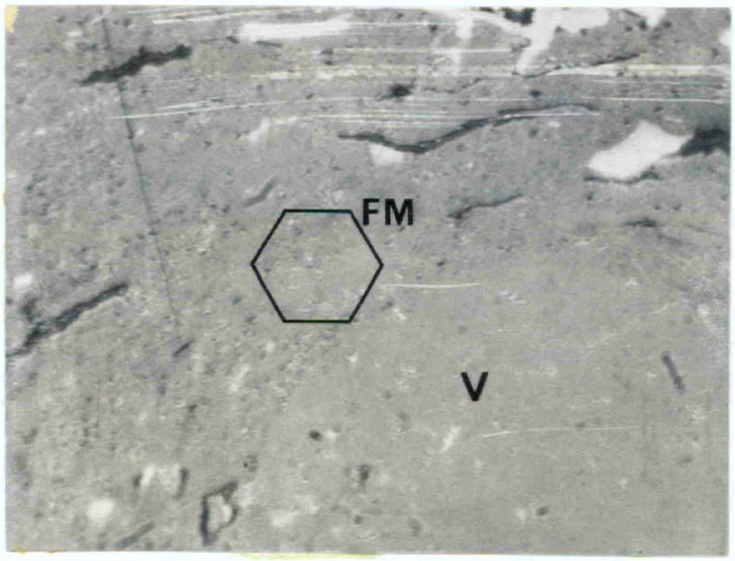

FIGURE 2 - Aumance Coal
V - structureless vitrinite; FM - vitrinite with fine micrinite, air (x 500)

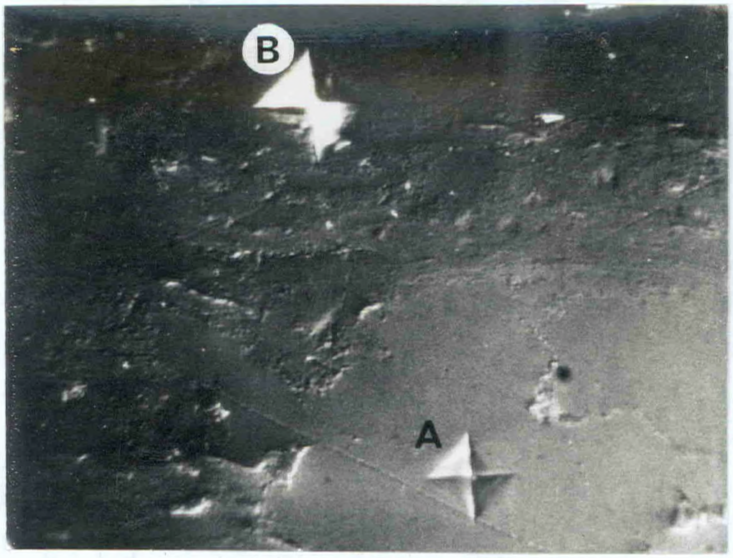

FIGURE 3 - Microhardness impression on original weathered coal; A - impression on structureless vitrinite; B - impression on vitrinite with fine micrinite; load 5 ponds, air (x 500)

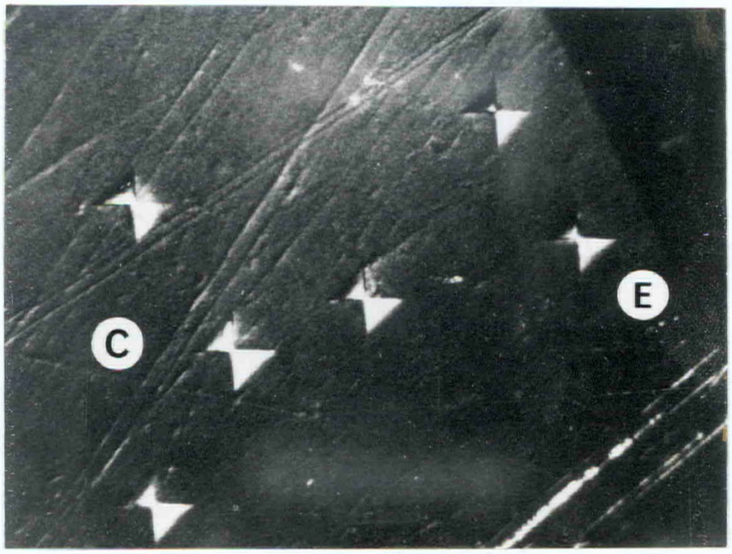

FIGURE 4 - Microhardness impression of weathered coal oxidized for 48 hours at $100^{\circ}C$. No change in impression from the edge to the centre, load 2.5 ponds, air (x 500).
E - edge; C - centre

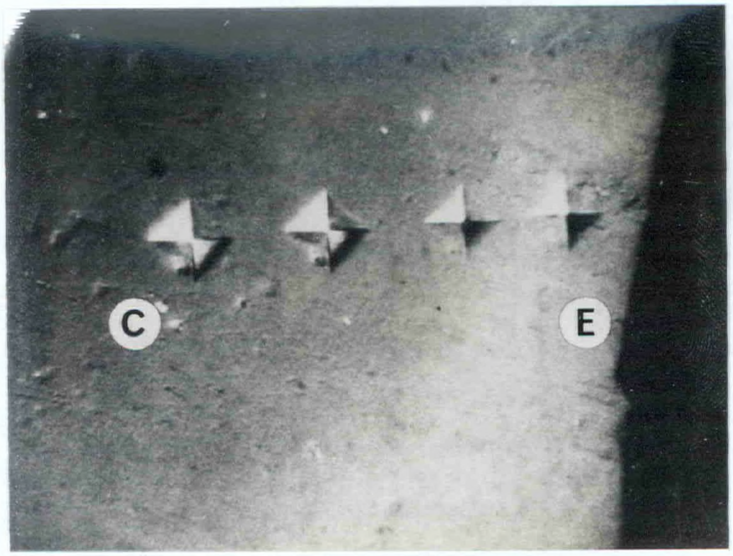

FIGURE 5 - Microhardness impression on weathered vitrinite (structured), oxidized under infrared lamp for 1.5 hours, load 2.5 ponds, air (x 500). E - edge; C - centre

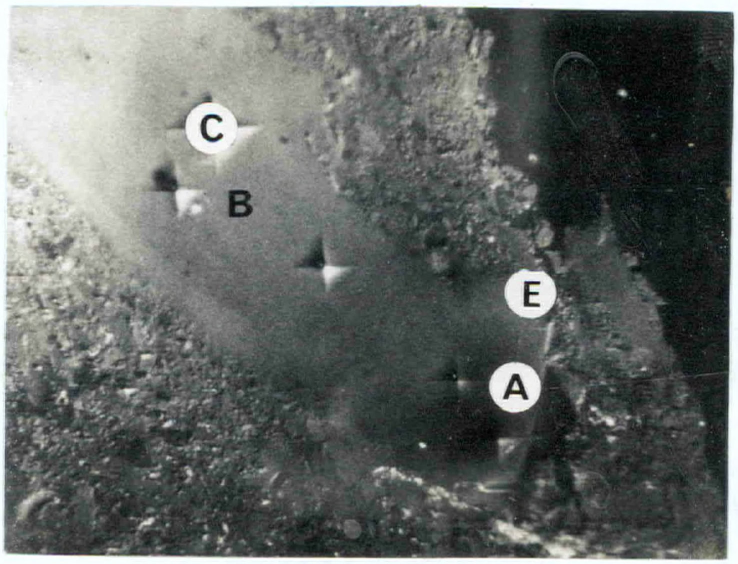

FIGURE 6 - Microhardness impression on weathered vitrinite (structureless) oxidized under infrared lamp for 1.5 hours, load 2.5 ponds, change in the impression elastic A from plastic B, air (x 500). E - edge; C - centre

FIGURE 7 - Dendritic dark structure of telinite in Bokaro Coal, air (x 500).

FIGURE 8 - Microhardness impression on weathered Ranigunj coal, elastic impression on the structureless vitrinite at E - edge, load 2.5 ponds, air (x 500).