TEMPERATURE-INDUCED CHANGES IN MAGNETIC SUSCEPTIBILITY OF PITTSBURGH COAL SEAM STRATA[1]

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Abstract: U.S. Bureau of Mines researchers are investigating geophysical methods for the remote detection of fires in abandoned coal mines and waste banks. All phases of fire control projects need inexpensive, noninvasive techniques for rapid and reliable evaluation of subsurface combustion location and extent. Heat from combustion causes mineral phase changes, altering the remanent magnetization and magnetic susceptibility of iron-containing minerals in carbonaceous strata. Paramagnetic pyrite can oxidize to strongly magnetic magnetite or to other oxides of iron with smaller positive magnetic susceptibilities. The minerals become more detectable as magnetic properties increase. The Bureau's laboratory project determined correlations between temperature and magnetic susceptibility for samples of coal mine roof strata from core drillings in the vicinity of a burning portion of an abandoned Pittsburgh seam mine in southwestern Pennsylvania. The magnetic susceptibility of each sample was measured before and after each trial at a furnace setpoint temperature (150°, 300°, 440°, 600°, 660°, 825°, or 980° C). Fifteen 20-to-25-g samples (triplicates of five carbonaceous materials) were placed in ceramic combustion boats; the heating rate was 60° C/h with a 3-h hold at the setpoint value. Oxygen concentration also affects changes in magnetic susceptibility. The furnace atmosphere source gas consisted of about 0%, 1%, or 10% oxygen in nitrogen, flowing at 1.4 to 2.4 L/min. Mossbauer spectroscopy and x-ray diffraction spectrometry identified iron compounds in the samples. Changes in magnetic susceptibility occurred as functions of both temperature and oxygen concentration.

Introduction

Researchers of the U.S. Bureau of Mines have been investigating methods for remote detection of underground fires in abandoned coal mines and waste banks. Residential, business, or recreational structures and their occupants can be adversely affected by the fumes or subsidence associated with the hidden combustion. Determining the location and extent of subsurface combustion is critical to minimizing the time, cost, and surface disruption of fire control and/or extinguishment projects. This assessment usually is done by evaluators standing on the surface, as much as several hundred feet above the combustion activity. Often, the extent of burning is estimated by observing surface venting locations and looking at available mining maps. There is a growing awareness of the need to apply sensitive and cost-effective diagnostics to determine accurately the extent and level of combustion in an inaccessible mine before control measures are initiated. Inexpensive, noninvasive techniques are also needed for use during and after control actions. Altogether, there is a need for more diagnostic alternatives, applicable to different situations, that will obtain sufficient information for choosing appropriate control actions while minimizing time and cost. Investigators have been trying various remote detection techniques to locate subterranean anomalies, including combustion, and enable them to map these phenomena. They have used electrical conductivity, electrical resistivity, ground penetrating radar, aerial photography (visible and infrared), seismometry, and boreholes (for corings, temperature measurements, and/or gas sampling). Magnetometry is one of the geophysical techniques being evaluated.

Magnetometry has had limited use as a remote detection method for establishing locations of underground fire zones. Magnetic anomalies have been associated with combustion metamorphism of oil-bearing sediments and burning or burnt Western U.S. coal-associated strata (Castleberry 1979, Cisowski and

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Fuller 1987, Geissman et al. 1983, Hooper 1987). Heat from combustion causes mineral phase changes that alter iron-containing materials in the overlying strata, affecting their remanent (permanent) magnetization intensities and magnetic susceptibilities.[3] Heating of these materials can increase their magnetic properties, thereby causing them to become more detectable. Therefore, the effects of underground combustion should be detected as changes in the intensity of magnetization measured above ground. One procedure for detecting subterranean magnetic materials uses the portable proton precession magnetometer (Breiner 1973). A limited magnetometer survey study of a burning abandoned underground coal mine site in Pennsylvania was done in 1988 and partly repeated in 1989 (Schueck 1990). Visual observations along the outcrop areas that had burned and cooled were correlated with positive magnetic anomalies (i.e., local increases in magnetization). These deductions and detected changes in magnetic intensity in the (inaccessible) mine workings permitted mapping of magnetic intensities relative to background. The map provided some idea of the progress of the fire zones after a year. Some correlations of magnetic properties of pyrite in coal with temperature and air partial pressure changes have been made (Thorpe et al. 1984). Correlations of magnetic properties of coal mine roof strata with temperature and oxygen concentration are not available in the literature. This paper describes a laboratory project to determine the effect of temperature on the magnetic susceptibility of strata components associated with the Pittsburgh coal seam. The results of this work should contribute to improving remote detection and delineation of underground abandoned coal mine fire zones using magnetic methods.

Experimental Approach

Laboratory studies were conducted on coal and roof shales from core drillings obtained in the vicinity of a burning portion of an abandoned underground Pittsburgh coal seam mine in southwestern Pennsylvania. These strata contain iron compounds that when heated change chemically with consequent alterations of their remanent magnetization. The primary independent variables are considered to be temperature and oxygen concentration because mine fires can smoulder at very low oxygen levels and exhibit local temperature increases owing to the low thermal conductivity of rock strata.

Portions of the core materials were pulverized so all pieces passed through a 2.36-mm dry sieve mesh. Each portion was divided into a minimum of nine representative samples; the remainder was stored in a glass jar. Each sample was weighed prior to the susceptibility measurement. The approximately 20-to-25-g, 15.5-cm^3 samples were put into cylindrical plastic containers that fit into the measuring coil of the magnetic susceptibility meter (Model SI-2, Sapphire Instruments, Canada).[4] The meter was controlled with a personal computer that recorded the magnetic susceptibility values of the samples in each test (fig. 1). The magnetic susceptibility of each sample was measured before and after heating in a tubular combustion furnace (fig. 2). Furnace setpoint temperatures were 150°, 300°, 440°, 600°, 660°, 825°, and 980° C. The heating rate was 60° C/h with a 3-h hold at the setpoint value. Each sample was poured into a ceramic sample boat (5.5 by 8.3 by 1 cm) able to withstand temperatures to 1,000° C. Five triplicate samples were randomly placed in the furnace at the beginning of a test series. The placement was retained for that series. After 3 h at the setpoint temperature in each test, the furnace's power was turned off and the equipment was allowed to cool prior to removal of the samples. A thermocouple at the center of the furnace was located between firebricks that provided horizontal support for the sample dishes. The brick temperature was expected to be closer in value to the sample temperature than was the gas temperature measured above the samples. The brick temperature attained equilibrium more slowly than did the setpoint and gas temperatures; this was the primary reason for the 3-h hold at the setpoint temperature. While both brick and gas final temperatures were less than the setpoint value,

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[3] The equivalent terms remanent magnetization and permanent magnetization are applied to rocks and to metals, respectively (Breiner 1973). Magnetic susceptibility is the measure of how easily a material can be magnetized. It is a ratio of the magnetic moment per unit volume to the product of applied magnetic field and the density of a substance (Senftle and Thorpe 1987).

[4] Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.

the brick value was at least 95% of the setpoint value from 440° to 980° C.

Furnace atmosphere was varied to determine any correlation between oxygen concentration and magnetic susceptibility of the heated carbonaceous materials. Approximately 0%, 1%, or 10% oxygen (O_2) in nitrogen (N_2) source gas was used in each test series. The gas flowed through the furnace at a rate of 1.4 to 2.4 L/min. An activated carbon filter installed at the vent end of the furnace adsorbed organic vapors from the heated materials. A particulate filter was placed between the vapor filter and the outlet flowmeter. Representative portions of the unheated samples were analyzed for their moisture, ash, volatile matter, fixed carbon, total iron, and forms of sulfur concentrations. Solids analyses using Mossbauer and x-ray diffraction spectrometry determined initial iron mineralogy and phase changes associated with heating.

Figure 1. Magnetic susceptibility meter and personal computer system with sample container.

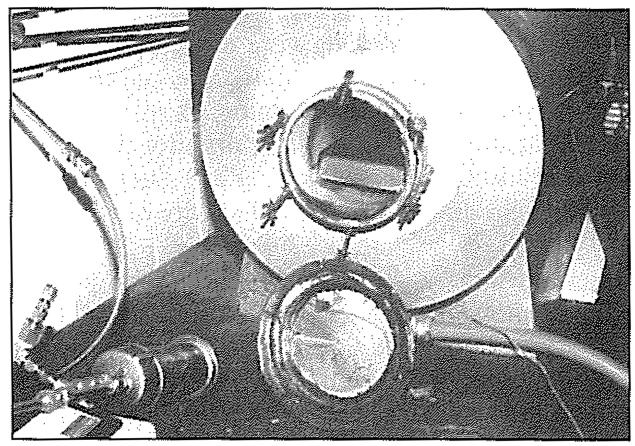

Figure 2. Tubular combustion furnace.

Two initial sets of heating tests with N_2 used furnace temperatures of 150°, 600°, and 980° C. For these samples, the data indicated significant increases in magnetic susceptibility at 600° C and subsequent decreases to near original values at 980° C. Four more temperature setpoints (300°, 440°, 660°, and 825° C) were added to complete the test protocol. Literature values for expected iron-containing compounds were considered when choosing the seven setpoint values. The susceptibility values at 150° C in the initial two trials had not changed from the ambient value. The first setpoint in the third set (also using N_2) was 300° C, whereas all subsequent sets began at 150° C. The third through fifth trials were run as identically as possible, each set with a different O_2 concentration. The following discussion and comparisons pertain to the three sets with the added setpoints and furnace source gas O_2 concentrations of about 0%, 1%, and 10%, respectively.

Discussion of Experimental Results

Gas sample analyses indicated that the lowest oxygen concentration attained at the furnace vent was 0.1% in tests conducted in nitrogen. It is possible that the "residual" oxygen desorbed from the firebricks used as props in the ceramic furnace tube or from the tube itself, or was generated during decarboxylation reactions in the heated carbonaceous matter.

Figure 3 depicts how the magnetic susceptibility of the material heated in N_2 (averaged over all the samples in the set) varied with temperature. These data were taken as the baseline for evaluating the effect of oxygen concentration, although there may have been as much as 1,000 ppm of O_2 in the furnace gas during heating. While there was a small positive change in magnetic susceptibility from the ambient temperature value to that at 300° C, there was an increase of greater than one order of magnitude when the temperature was raised to 440° C. The decrease in magnetic susceptibility as temperature increased to 825° C implied that magnetically weaker mineral phases were being formed. The magnetic susceptibility increase at 980° C to between the average values at 600° and 660° C is interesting because it indicates that magnetically stronger phases were again formed.

Both temperature- and oxygen-related changes in magnetic susceptibility are shown in figure 4. There were significant increases, from the values at the next lower setpoints in each set, at both 440° and 980° C for both 1% and 10% O_2 sets, as noted for the N_2 atmosphere data. Except at the 440° C setpoint, the 1% O_2 data values were greater than those of either of the other two sets. It can be inferred that at the 10% O_2 concentration, the oxides formed were magnetically weaker than those formed in the 1% O_2 atmosphere. These results may be partly explained by comparing the quantities of iron (Fe) and O_2 available during a heating trial. The material in each of the sample boats (18 to 27 g) contained a low percentage of total iron (3% to 14% in the original samples) for a maximum of 1.02 mol Fe per test. The flow of gas (1.4 to 2.4 L/min) through the furnace provided a (minimum) total of 0.11 mol O_2 in 1% O_2 and 1.1 mol O_2 in the 10% O_2 tests.

A different way of viewing the data to determine how the magnetic susceptibility changes could affect field measurements is depicted in figure 5. The average ratios of magnetic susceptibility values (after heating to unheated) were plotted versus the various setpoint temperatures for each set of oxygen concentrations. The maximum ratio, at 600° C and 1% O_2, was about 128 times the unheated magnetic susceptibility. For individual sample ratios between 440° and 980° C at 1% O_2, the smallest ratio was about 15 (at 440° and 825° C) and the largest was about 174 (at 600° C).

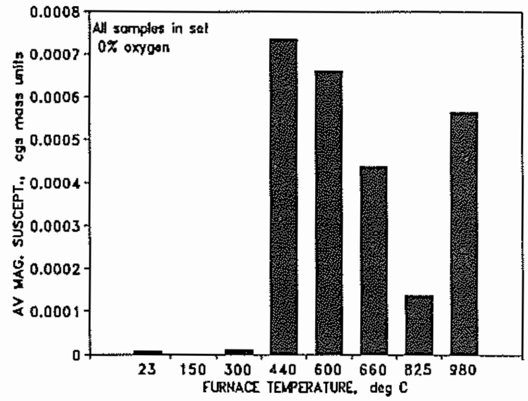

Figure 3. Average magnetic susceptibility (15 samples) versus temperature in nitrogen atmosphere.

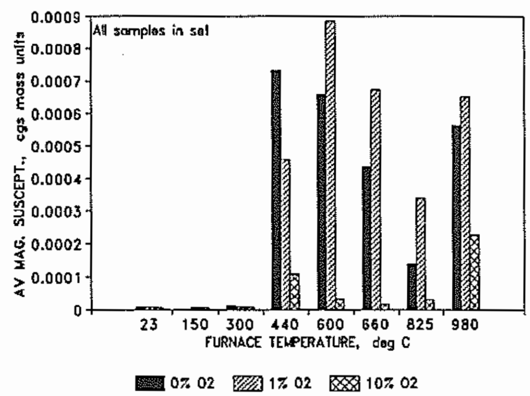

Figure 4. Average magnetic susceptibility (15 samples per set) versus temperature for three oxygen concentrations.

Summary

Laboratory experiments show that there is a thermal effect on the magnetic susceptibility of Fe-containing minerals in carbonaceous strata overlying the Pittsburgh coal seam. Oxygen concentration during heating also affects the magnetic susceptibility. Magnetically stronger mineral phases apparently formed at about 440° C and again when the materials were heated to about 980° C; ongoing solids analyses should define the phases produced at the several temperature and oxygen conditions. The experimental data indicate that the increase in the magnetic susceptibility after heating of the carbonaceous materials is less at 10% O_2 than at 1% O_2, but the increase over the value at ambient temperature is significant in either case. The inference is drawn that sensitive field measurements of another magnetic property of iron-containing materials-the magnetic intensity-may allow remote detection for detailed mapping of both burning and burnt coal. The Earth's magnetic field in an area is sufficiently constant that sensitive magnetometers could differentiate among the magnetic intensity levels it induces in unburnt, burning, and burnt coal seam strata.

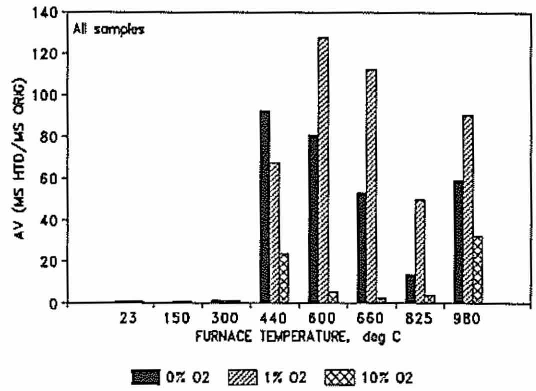

Figure 5. Average ratio of magnetic susceptibilities: heated/original versus temperature for all samples in each set.

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