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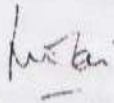
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PREFACE

The Second International Conference on Advanced Materials Processing (ICAMP 2002) was a great success, with two key note and 9 invited addresses and over 130 oral presentations by delegates from over 20 countries. The three-day conference at Grant Hyatt Hotel, Singapore, was packed with a wide range of discussion topics from thin films, nano-structured materials, advanced metal-matrix composites, light weight alloys and functional materials to characterization, laser materials processing, ceramics processing, and modeling and simulation. The papers presented during the conference are now included in these proceedings in their full lengths. I hope that these papers will provide stimulus for future advancement in materials research for the benefit of mankind.



M.O. Lai
Conference Chairman
ICAMP 2002

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Abstract. Magnetic properties of coal samples were studied by using electron spin resonance (ESR) spectrometer. The spectra linewidth were determined from ESR measurement results. We observed that these parameters depend on heating temperature. Spectra of all samples showed both broadening and narrowing phenomena. It is indicated that the change of spectra linewidth is due to dipolar interactions of unpaired electrons and the unresolved hyperfine structure of interactions of unpaired electrons with neighbouring protons.

Introduction

The evolution of free radical system in heated coal can be studied by electron spin resonance [1-6]. Retcofski et al. [2] presented a detailed study of the ESR linewidths of coals and concluded that unresolved electron-nuclear hyperfine interactions contribute significantly to the observed linewidths. This explanation is based principally on the smooth variation of linewidth with hydrogen content. Hydrogen is the most abundant element in coal having a nonzero magnetic moment and the most likely to interact with the free radical electrons in coal. The most important factors determining linewidth are hyperfine interactions, spin-lattice interaction, spin-spin interaction and exchange interaction.

The spectrum of polycrystalline or amorphous material will be smeared out to a single broad line, since the hyperfine interactions will be randomly oriented. When heating treatment is done up to 400°C, hyperfine interactions between the unpaired electrons and hydrogen nuclei make a role play for the linewidth. Spin-lattice interaction enables the magnetic energy, which has been absorbed by the unpaired electrons, to be converted into thermal energy. The most important process that contribute to spin-lattice interaction is spin-orbit coupling, which gives broadening linewidth. Spin-spin interaction involves the local magnetic fields of neighbouring unpaired electrons. The closer the neighbouring unpaired electrons, the greater spectrum linewidth.

Exchange interaction occurs when the freedom of movement of the unpaired electron is not restricted to a particular radical. The effect is to average out the local magnetic fields of neighbouring unpaired electron and to reduce the linewidth. A line narrowed by exchange is characterised by a Lorentzian lineshape [3]. Pilawa *et al.* [4] observed that dependence of the linewidth on temperature is caused by the changes in spin-spin and spin-lattice interactions of unpaired electrons. This is a result of the changed chemical structure of environment of free radical in macerals. Spectral linewidth for the lower rank material are between 1.9 and 8.0 gauss.

The linewidth of resonance lines is the direct result of spin-spin interactions between the free radical electrons and protons in the coal. The greater mobility of unpaired electrons associated with large aromatic ring systems may produce exchange narrowing of the resonance line. Unpaired electrons of multiring structures, mainly delocalized π -electrons with strong exchange interactions, are responsible for the narrow components of the resonance absorption spectra. Unpaired electrons of aliphatic structures or structures composed of a few aromatic rings are responsible for the broad lines. The high values of the linewidths of resonance signals can be caused by dipolar interactions of unpaired electrons and the unresolved hyperfine structure of interactions of unpaired electrons with neighboring protons [5].

The aim of the present work was to determine the changes of the behavior of free radicals during heat treatment. Linewidths and lineshapes of the first derivative absorption curves at the points of heat treatment were analyzed and compared to identify the interaction involved.

Experimental Method

Two samples of coals were purchased from Kalimantan, one sample from East Kalimantan (we abbreviate it as KTA) and the other one from South Kalimantan (we abbreviate it as KSA). All samples were powdered to a few microns in size and then filled in ceramic crucible. The powdered coals were heated in a Thermoline-6000 furnace in the range of heat treatment 30 – 300°C. At each points of the heat treatment, samples were held at the constant temperature for about one hour.

The measurements of ESR were made at room temperature, using a JES RE-2X spectrometer that operates at the frequency 9.4 GHz (X-band) at Physics Department, University of Indonesia. Before measuring ESR, the mass of all samples were measured and the sample were filled in quartz tube that will be held in the resonator cavity. The peak-to-peak linewidth, ΔH_{pp} , was obtained from the width between the maximum and the minimum point of the recorded resonance absorption curve.

Results and Discussion

Spectrum lineshape of all samples showed a single broad line because hyperfine interactions were randomly oriented. Figure 1 shows the influence of heat treatment of the sample on the linewidth of KTA-sample. The linewidth of KTA-sample at temperature 27 – 100 °C is decreased, due to decreasing hyperfine interaction between the unpaired electron and hydrogen nuclei. The decreasing linewidth at temperature 150 – 300 °C can be explained by the increasingly narrowing effect of exchange interactions of the unpaired electron of multiring structure, mainly delocalized π -electron. The increase of linewidth at temperature 100 – 150 °C can be explained by the increasing dipolar interactions of unpaired electron of aliphatic structure or structures composed a few aromatic ring. The proton-broadening hypothesis is also supported by a comparative ESR study of coals and dehydrogenated coals [5]. The dependence of the linewidth on temperature is caused by the changes in spin-spin and spin-lattice interactions of unpaired electrons.

Figure 2 shows the influence of heat treatment of the sample on the linewidth of KSA-sample. The linewidth sample at temperature 27 – 200 °C is approximately constant, whereas at temperature 200 – 300 °C is decreased. The decreasing linewidth is due to the increasingly narrowing effect of exchange interactions, mainly delocalized π -electron. Electron exchange narrowing can result from free electron transfer from one aromatic site in coal to another. For electron exchange frequencies less than the hyperfine splitting constants, the resulting band of unresolved hyperfine lines is Gaussian, whereas more rapid exchange results in a Lorentzian band. The lineshapes of coals are closer to Lorentzian than Gaussian and it may be concluded that the results are consistent with exchange narrowing of the ESR resonance [4].

Summary

We observed that linewidth of resonance absorption spectra depends on heating temperature. Linewidth showed both broadening and narrowing phenomena. The narrowing phenomena are suggested due to strong exchange interactions among free radicals of multiring structure of delocalized π -electron. While the broadening phenomena is suggested due to free radicals of aliphatic structure or structure composed of a few aromatic rings. We also investigated that the dipolar interaction and hyperfine structure are considered to play an important contribution to the linewidth.

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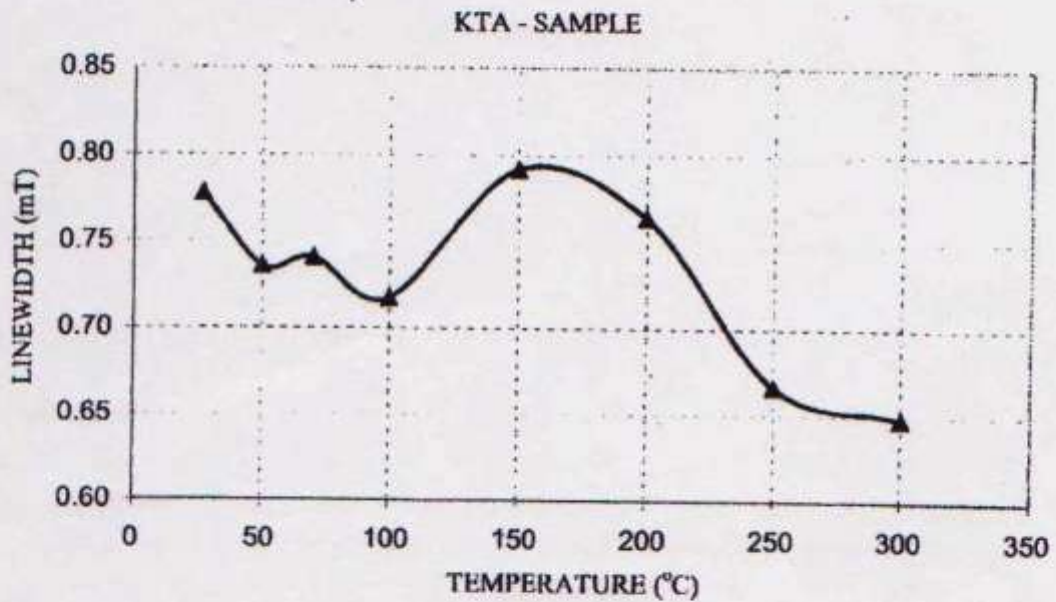


Figure 1. The linewidth vs heat treatment temperature of KTA-sample

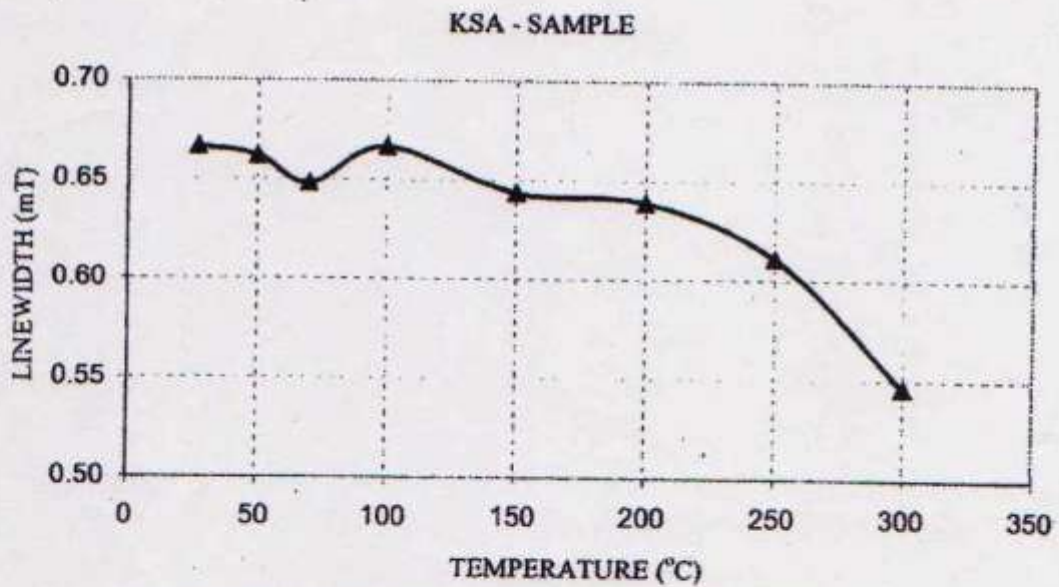


Figure 2. The linewidth vs heat treatment temperature of KSA-sample