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January 1985

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PITCH MIXED WITH TRINITROPHENOL**

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ABSTRACT

When coal-tar pitch is mixed with trinitrophenol (TNP) and carbonized, the formation of mesophase in the pitch is totally suppressed, and a porous honeycomb-like isotropic hard carbon is formed, which does not graphitize when heated at 2500°C for one hour, and only partially graphitizes when heated at 3000°C. The unmodified pitch coke readily graphitizes when given the same heat treatment. X-ray data show that at 3000°C the modified pitch coke consists of two components with $d_{002} = 3.36$ and 3.42\AA , respectively. An ESR spectrum is observed at all stages during the heat treatment of the

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modified pitch coke, even in the range 1000-2000°C where the spectrum of the pitch coke disappears. Two components are also observed in the ESR spectrum of the modified pitch coke at 3000°C. When logarithms of unpaired electron concentrations are plotted against values of Δg ($\Delta g = g - 2.0023$), a linear dependence is found in the HTT range from 300 to 3000°C in both cokes.

1. INTRODUCTION

The carbonization and graphitization of coal-tar pitch mixed with mono and dinitro compounds have been studied by Yamada [1]. His study is worth noting because it showed that: (a) the dehydration reaction between coal-tar pitch and nitro compounds proceeds at an early stage of pyrolysis, and (b) the dehydration reaction brings cross-linking into the structure. The cross-linking is so complicated that little graphitization occurs even at high heat-treatment temperature.

We are interested in the modified pitch coke obtained by addition of polynitro compounds to coal-tar pitch in connection with mesophase formation during carbonization. Unless the mesophase appears during carbonization the carbon thus obtained will not graphitize. There does not appear to have been any previous report concerning mesophase formation during the carbonization and graphitization of polynitro compound/coal-tar pitch mixtures. Moreover, in the previous work the characterization of the structural

changes during heat treatment has been limited mainly to X-ray analysis.

In this paper, we will study the carbonization of coal-tar pitch mixed with TNP and the structural changes of this modified pitch coke when heat-treated up to 3000°C by X-ray diffraction, optical microscopy, and ESR measurements, and compare the results with those obtained with unmodified pitch coke.

2. EXPERIMENTAL

The properties of the coal-tar pitch used in this work are shown in Table 1. The 2, 4, 6-trinitrophenol (TNP) used was commercial reagent grade (mp 121.5-122°C) and was dried before use. The trinitrotoluene (TNT) used for comparison with TNP was also commercial reagent grade (mp 80°C). The 2, 2-diphenyl-1-picrylhydrazyl (DPPH) used as the standard sample for ESR measurements was purified by recrystallization twice from benzene, and was dried under reduced pressure at 80°C (mp 139-140°C).

The coal-tar pitch and polynitro compounds were mixed in various proportions and finely ground in a mortar before heat treatment. Heat treatments up to 1000°C were performed on 1-2 g of each sample under an atmosphere of flowing argon gas. At heat-treatment temperatures (HTT) above 1200°C, the pitch cokes were placed in a small graphite container and heat-treated to the desired temperature in an electric graphite tube furnace in pure argon atmosphere. Each heat treatment was conducted at a heating rate of 120°C/hr up to

1000°C and held for one hour at maximum temperature. Above HTT 1200°C, each heat treatment was carried out at a rate of 295°C/hr and held for half an hour. Temperatures were measured by observation of the surface of the graphite container with an optical pyrometer which was calibrated beforehand. Weight loss (%) was determined from measurements of the change in weight before beginning and after the completion of a run.

The X-ray diffraction profiles of the (002) line of the powdered samples were measured with Ni-filtered CuK_α radiation. The d_{002} interlayer spacing was determined by reference to an internal standard of silicon, and L_c parameters were estimated from the (002) linewidth after correction in the usual way [2].

The ESR measurements were carried out with a JEOL X-band spectrometer, model JES-PE-1X, using 100 KHz magnetic field modulation. The g values were measured by comparison with Mn^{2+} in MgO placed in the cavity close to the sample position. The resonance conditions for the standard sample and the test sample are given by the equations:

$$\begin{aligned} \text{standard: } h\nu &= g_1 \beta H \\ \text{sample: } h\nu &= g\beta (H - \Delta H) \end{aligned}$$

Here ν is the frequency of microwave, g is the g value, β is the Bohr magneton, H is the field at resonance for the standard sample, and ΔH is the difference between the field at resonance for the test

sample and H. The change of field at resonance, ΔH , between these two samples was determined from the change of position of the center of the resonance on the recorder traces. Thus, the g value of the sample was calculated using

$$g = \frac{g_1 H}{H - \Delta H}$$

The g value of Mn^{2+} was determined beforehand by comparison with the value of DPPH ($g = 2.0036$). The unpaired electron concentration of each sample was estimated from the area of the absorption spectrum based on that of DPPH. The area S was estimated from the equation $S = \Delta H^2 \cdot h$, where ΔH is the line width and h is the peak-to-peak height of the derivative spectrum. This approximation was used because the shape of the observed spectra after carbonization was almost Lorentzian [3]. For HTT below $400^\circ C$ the absorption line shape was different from the Lorentzian line shape, but the unpaired electron concentration estimated using $S = \Delta H^2 \cdot h$ did not differ appreciably from that estimated from the double-integrated absorption intensity of the first-derivative spectrum. The temperature for ESR measurements was controlled by a standard JES-VT-3A variable-temperature controller. Prior to recording an ESR spectrum the heat-treated sample was ground, put into a standard sample tube, heated at $150^\circ C$ for several hours and then sealed after evacuation by a vacuum-line system ($< 10^4$ Torr).

Optical observations were made on the surface of samples which had been mounted in a resin, cured and polished with SiC abrasives followed by 0.5 μm Al_2O_3 powder to obtain a mirror finish.

3. RESULTS

Yield and Optical Observations

Figure 1 shows the weight loss for the coal-tar pitch and the coal-tar pitch/TNP mixture versus HTT. It can be seen that the weight losses are far smaller from the mixture than from the pitch coke alone. Similar curves were obtained from the other mixtures. Figure 2 shows the yield of the modified pitch coke at HTT 1000°C versus the coal-tar pitch/TNP weight ratio (Curve (a)), and also shows the calculated carbon contents versus mixing ratio (Curve (c)). It can be seen that the yield of the modified pitch cokes initially increased with increase of TNP content, and a maximum yield was obtained from the 80% coal-tar pitch/20% TNP mixture. At this mixing ratio, the carbon in the coal-tar pitch was fixed in the largest amount. (The yield was about 70%, whereas the calculated carbon content before carbonization was 79.8%.) A similar curve of yield versus mixing ratio for coal-tar pitch/TNT mixtures is also seen in Fig. 2 (Curve (b)). It appears that the increase of the yield of the coke obtained from a mixture of coal-tar pitch and aromatic polynitro compounds is a common phenomenon. TNP has a larger effect of fixing the carbon in the coal-tar pitch than does TNT. The 80% coal-tar

pitch/20% TNP mixture was chosen for a starting sample in all further experiments in this work.

On heating above their melting points the coal-tar pitch/TNP mixtures did not melt completely, and began to foam at HTT about 200°C. The foaming was vigorous if the heating rate was rapid. During this foaming, condensation of water vapor could be seen on the sight window in a closed system.

Optical micrographs, Figure 3, show sections of a foamed fragment of modified pitch coke for several HTT. Many small pores were present at 200°C, they became smaller and the number diminished at HTT 300°C, and they could not be seen at HTT 500°C. It is known that the disappearance of these pores occurs during the carbonization of thermosetting polymer resins which convert to glass-like carbons [4-6]. When the sections of modified pitch coke shown in Fig. 3 were viewed under polarized light, no anisotropic area could be observed. After heat treatment at 2500°C, the modified pitch coke is completely isotropic as shown in Fig. 4(a). On the other hand, when the coal-tar pitch was heat-treated at various temperatures, regions of mesophase appeared at HTT 450°C and a change to bulk mesophase occurred at HTT 600°C. After HTT 2500°C, the bulk mesophase changed to the flow-type texture as shown in Fig. 4(b). That is, an anisotropic texture was maintained after high temperature heat-treatment.

X-Ray Diffraction

Typical (002) X-ray diffraction profiles of the modified pitch coke heat-treated at various temperatures are shown in Fig. 5. The profiles are very broad up to HTT 2500°C. After heat treatment at 3000°C, the profiles narrowed and split into at least two components. The composite profile can be separated into two components as shown in Fig. 6. From this figure d_{002} and L_c of the components were estimated. Component A had d_{002} of 3.360Å and L_c greater than 1000Å, whereas component B had d_{002} of about 3.42Å and L_c of about 60Å. The (002) diffraction profile of the pitch coke up to HTT 2000°C was always much sharper than that of the modified pitch coke, and contained only one component. That is, the pitch coke was more easily graphitized. The changes of d_{002} and L_c of pitch coke with HTT are shown in Fig. 7. The value of L_c increased sharply after d_{002} decreased below 3.425Å.

ESR Measurement

The unpaired electron concentration as a function of HTT is shown in Fig. 8. The unpaired electron concentration in the pitch coke and in the modified pitch coke increased as the carbonization reaction proceeded, attained a maximum in the HTT range 600-800°C, and thereafter decreased to HTT 1000°C. In the HTT range 1000-2000°C, the ESR spectrum of the pitch coke could not be observed, but a spectrum was observed throughout this range for the modified pitch coke. It is worth noting that the unpaired electron concentration of

the modified pitch coke increased in this HTT range. For the pitch coke heat-treated at 2000°C, the ESR spectrum appeared again and the unpaired electron concentration gradually decreased with increasing HTT as in the case of the modified pitch coke. Figure 9 shows the ESR spectrum of the modified pitch coke heat-treated at 3000°C measured at 20°C and at -160°C. It was assumed that two components, broad and narrow, are present in each spectrum. By comparing the intensities the broad-line component was found to be more affected by measurement temperature than the narrow one. The change of Δg ($\Delta g = g - 2.0023$) with HTT is shown in Fig. 10. This change was contrary to the change of unpaired electron concentration with HTT. That is, the Δg decreases with the increase of unpaired electron concentration. Near the maximum point of unpaired electron concentration, the Δg showed negative values, i.e., the g value becomes smaller than the value of free spin, $g = 2.0023$.

4. DISCUSSION

The results show that mesophase formation during the carbonization of coal-tar pitch was suppressed by mixing with TNP. The observed foaming and evolution of water vapor indicate that cross-linking occurs at HTT as low as 200°C. This agrees with the earlier observation that a dehydration reaction occurs between mono and dinitro compounds and coal-tar pitch at an early stage of pyrolysis as shown by Yamada [1]. As the dehydration reaction proceeds, the cross-linked structure is formed, and this prevents the

appearance of liquid phase. Moreover, this cross-linking reaction brings a high yield of coke as shown in Fig. 1. Presumably instead of evaporating, small hydrocarbon molecules assist in producing the cross-linking. The 80% coal-tar pitch/20% TNP mixture appears to be optimum for building a three dimensional network from these components. However, not only the nitro groups but also the hydroxyl group in TNP works to build the network. This is supported by the observation that the yield of coke obtained from the coal-tar pitch/TNP mixture is higher than that from the coal-tar pitch/TNT mixture as shown in Fig. 2.

The X-ray data, Fig. 5, clearly show that the modified pitch coke does not graphitize up to 2500°C HTT. Although it is possible to make a separation into two components at 3000°C HTT, the origin of the narrow component is not clear. It may be that, owing to increasing crystallite size a phase present at lower HTT has provided an intensity just large enough to measure. However, it seems more likely that the narrow component is due to a partial stress graphitization. It is well known that when mixture of hard carbon, e.g., glassy carbon, and pitches are heat-treated above 2000°C, graphitization of the hard carbon phase goes to completion readily. Thus, if a second phase were present at lower HTT, the modified pitch coke would have graphitized at 3000°C.

In view of the above, it is natural to interpret the ESR data of the modified pitch coke by comparison with data obtained from heat-treated hard carbons. In another investigation, to be reported

later, it was found that the ESR data obtained from the modified pitch coke in the present work is similar in all respects - line shape, temperature dependence, the maximum value of N - to those obtained from a variety of glassy carbons.

The processes of carbonization and graphitization of the two pitch cokes can be correlated with the unpaired electron concentration. They can be separated into three stages.

The first stage corresponds to the region of HTT up to 1000°C. In this stage the unpaired electron concentration increased up to a maximum in the HTT range 600-800°C and thereafter decreased. This behavior is commonly observed during the carbonization reaction and the maximum value of N attained, which corresponds to about 10^{-3} unpaired electrons per carbon atom, seems to be independent of the precursor hydrocarbons. The unpaired electron concentration probably increases because impurity atoms - hydrogen, oxygen, nitrogen - are removed from the hydrocarbons in the coal-tar pitch and TNP leaving dangling bonds. The decrease is attributed to the synthesis of larger aromatic molecules from smaller ones. The unpaired electron concentration reaches a maximum in the region of the temperature where the carbonization reaction nears completion, which is supported by the observation in Fig. 1, that the weight loss was very small at HTT above 800°C. Also, it is well known that carbons experience very little weight loss above HTT 1000°C [8-9]. The decrease of the unpaired electron concentration seen at HTT above 800°C means that the rate of the disappearance of unpaired electrons by synthesis of

larger molecules is faster than the rate of removal of impurity atoms.

The second stage corresponds to the HTT range from about 1000 to 2000°C. This stage can be explained by assuming that at the completion of the first stage the structure consists of fused aromatic rings whose separation is maintained by interlayer carbon-carbon or carbon-hydrogen bonds. Heating above 1000°C removes these interlayer atoms. The X-ray data in Fig. 7 shows that the interlayer spacing in the pitch coke begins to decrease in this region and decreased strongly when HTT reaches 2000°C. Concurrently, the ESR spectrum could not be observed in the pitch coke heat-treated at 1250, 1500 and 1750°C. The disappearance of the ESR spectrum in the temperature range 1000-2000°C is well known for soft carbons, and a decrease in this range is also commonly observed in hard carbons. In general, it has been considered that the disappearance is an artifact, i.e., because the line width of the spectrum is too wide to be observed. Figure 11 shows the change of line width of the ESR spectrum of the pitch cokes heat-treated at various temperatures. If it is assumed that the line width continues to increase in the HTT range from 1000 to 2000°C, the ESR spectrum should not be observable under ordinary conditions. The line width of ESR spectrum is related to the spin-spin relaxation time T_2 , and becomes short according to the equation $T_2 = h/(\sqrt{3g}\beta\Delta H)$ [3]. However, the disappearance of the ESR spectrum of pitch coke in the HTT range of 1000-2000°C cannot be explained by the line-broadening only, because it can be enhanced by

changes of both sweep width and modulation of the ESR apparatus. This was tried without success in this experiment, even when the spectrum has a width greater than 500 gauss.

Another possible explanation for the disappearance of the ESR signal is the "silicon effect." Singer and Wagoner have shown that impurities such as SiO_2 , SiC , Si and Ge cause this phenomenon [10]. Their explanation is that Si or Ge or heavy elements diffuse into the sample rapidly at temperatures around $1000\text{-}1100^\circ\text{C}$, probably forming surface complexes which act as effective agents for spin-lattice relaxation. These impurities are driven out at about $\text{HTT } 1400^\circ\text{C}$ and above. This explanation was verified by purifying samples showing the "silicon effect" in flowing Cl_2 gas, after which the effect disappears, i.e., the ESR signal becomes observable. A more detailed study on the impurity broadening of the ESR spectrum has been made by Mrozowski [11]. In view of the fact that no purification treatments were given to the samples in this work, it is not possible to rule out the silicon effect as the cause of the signal disappearance. It seems likely that the same phenomenon is also present in the modified pitch coke, but to a lesser extent.

The third stage corresponds to the region of above $\text{HTT } 2000^\circ\text{C}$. In this stage no large scale rearrangement of layer planes occurs but the crystallite size increases. The decrease of the unpaired electron concentration in this stage is attributed to annealing of the remaining point defects.

The change of unpaired electron concentration with HTT shown in Fig. 8 is well known as a general behavior in carbons, and the peaks seen at HTT 600-800°C and around 2000°C are explained from a contribution of localized spins and mobile carriers [12,13]. However, there are few reports concerning the relation between the unpaired electron concentration and the g shift [14,15]. None have suggested a specific relationship. When the logarithms of unpaired electron concentration ($\log N$) were plotted against the values of Δg of pitch coke and modified pitch coke, a good linear relationship was established as shown in Fig. 12. That is, the empirical equation is given by

$$\log N = A + B \cdot \Delta g \times 10^4 ,$$

where A and B are parameters depending on the materials. From a linear least-squares analysis, $A = 19.2 \pm 0.1$ and $B = -0.05 \pm 0.002$ were obtained on the pitch coke and $A = 20.5 \pm 0.2$ and $B = -0.064 \pm 0.005$ were obtained on the modified pitch coke. There is little difference between A and B from both cokes. The above equation shows that in pitch coke and modified pitch coke the unpaired electron concentration can be estimated from a measurement of the g value.

In summary, the present work shows that when polynitro compounds are mixed with pitch coke the carbonization-graphitization behavior is similar to that found with mono and di-nitro compounds. That is,

a hard carbon is formed which is similar to glassy carbons formed by different synthesis procedures.

ACKNOWLEDGEMENT

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Table 1. Properties of Coal-Tar Pitch

Chemical composition, wt%

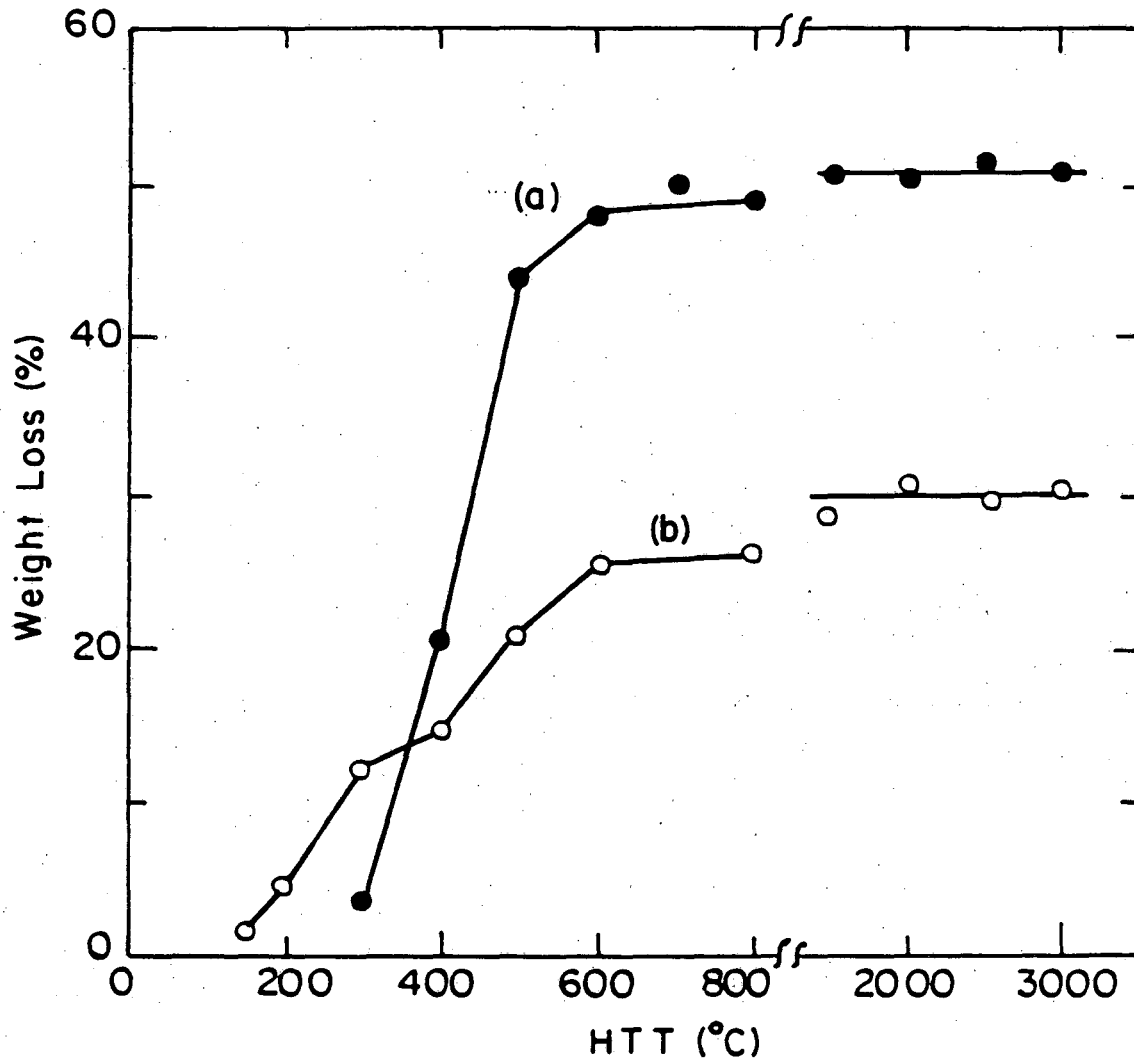
C	91.92
H	4.84
N	1.29
S	0.50
ash	0.02

Distribution for solvent, wt%

toluene insoluble	14.60
quinoline insoluble	0.57

Density 1.28 g/cm³

Softening point (R & B) 101°C

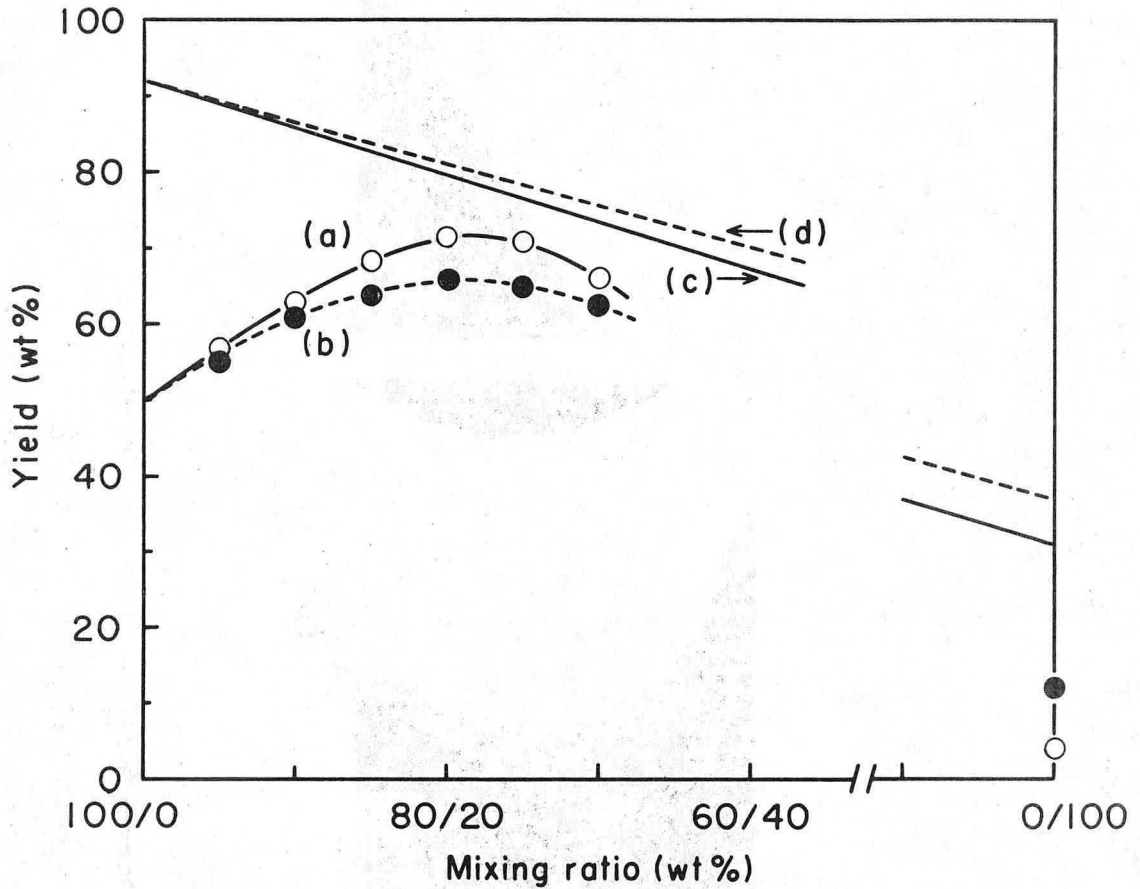


XBL 8411-6096

Fig. 1. Weight loss as a function of HTT.

(a) Coal-tar pitch

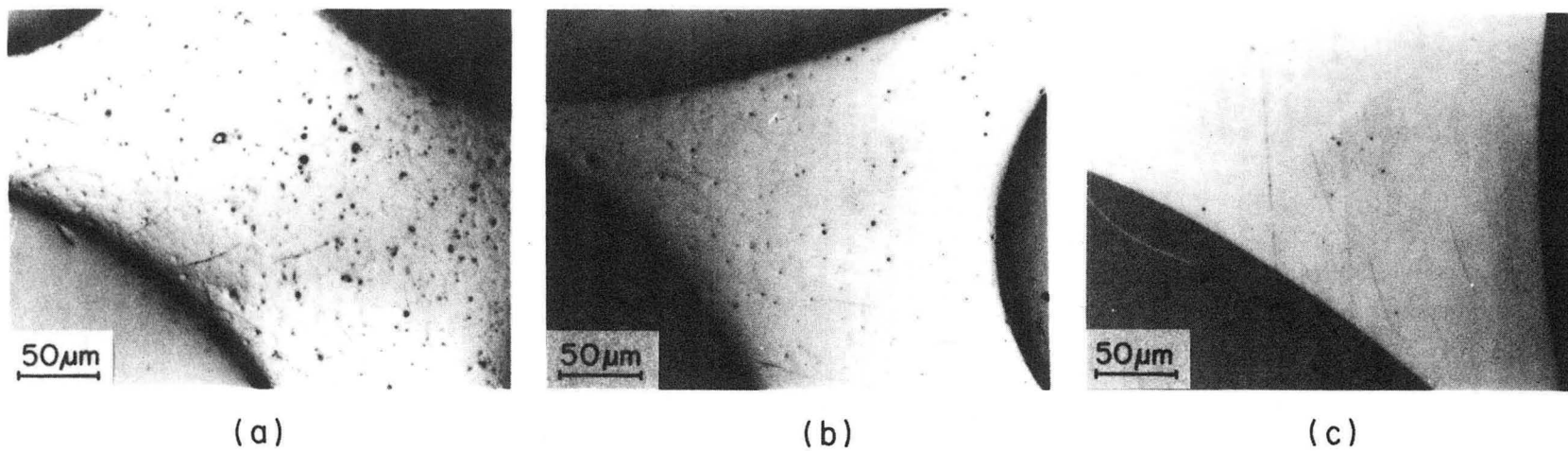
(b) Coal-tar pitch/TNP (80/20) mixture



XBL 8412-5866

Fig. 2. Comparison of yield and carbon content.

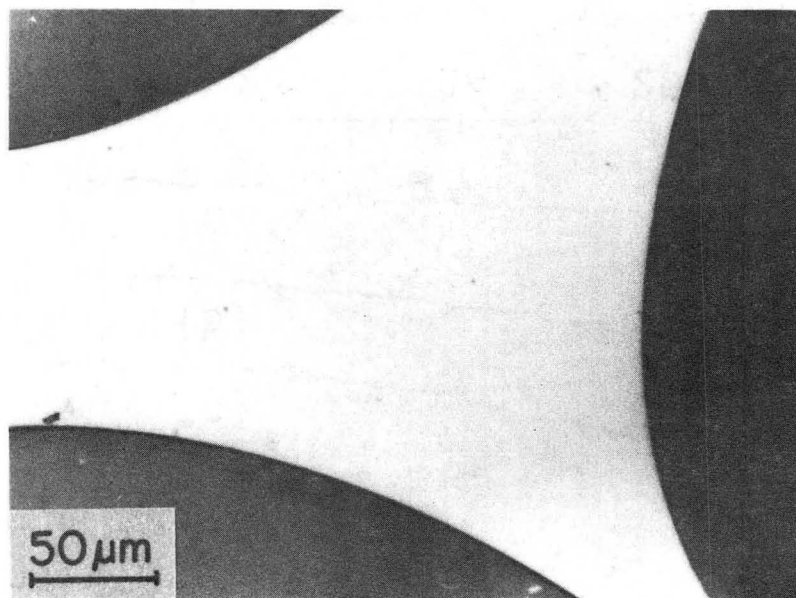
- (a) Yield of pitch coke modified by mixing of TNP at HTT 1000°C.
- (b) Yield of pitch coke modified by mixing of TNT at HTT 1000°C.
- (c) Calculated carbon content in coal-tar pitch/TNP mixture.
- (d) Calculated carbon content in coal-tar pitch/TNT mixture.



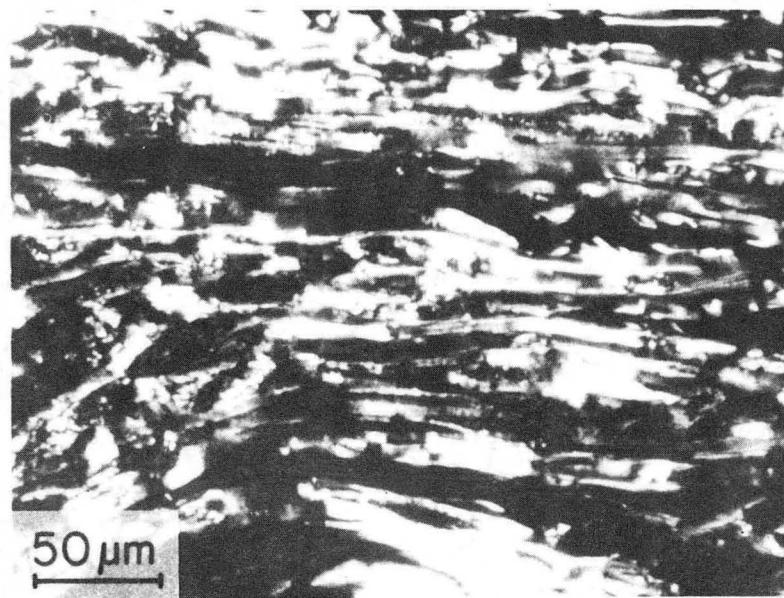
XBB 840-9437

Fig. 3. Micrographs of modified pitch coke by mixing of TNP.

- (a) Heat-treated at 200°C
- (b) Heat-treated at 300°C
- (c) Heat-treated at 500°C



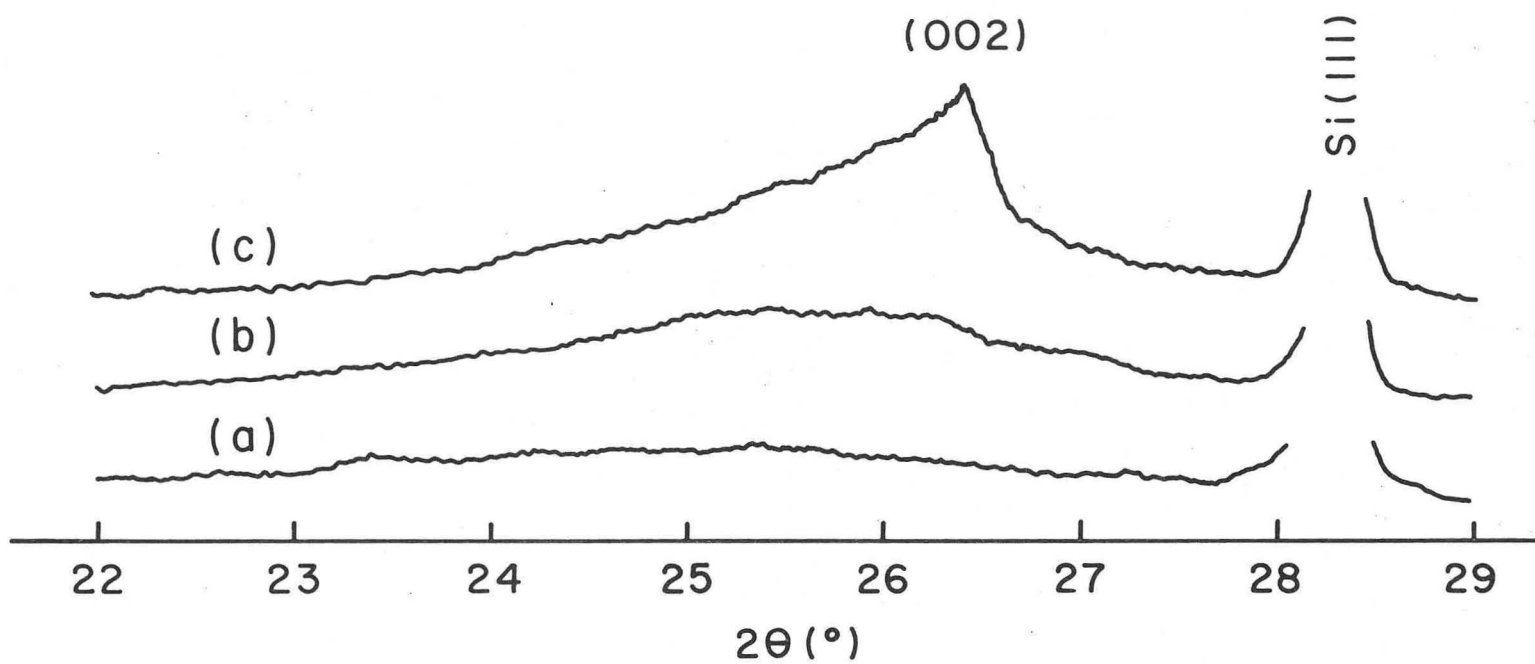
(a)



(b)

XBB 840-9438

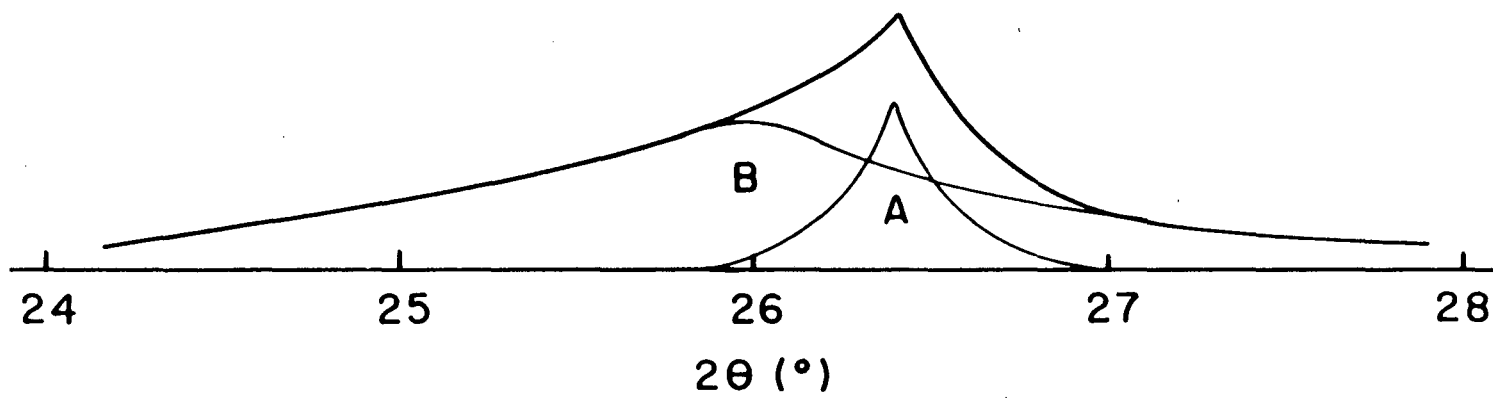
Fig. 4. Comparison of micrographs under crossed nicols of (a) modified pitch coke and (b) pitch coke heat-treated at 2500°C.



XBL 8412-5867

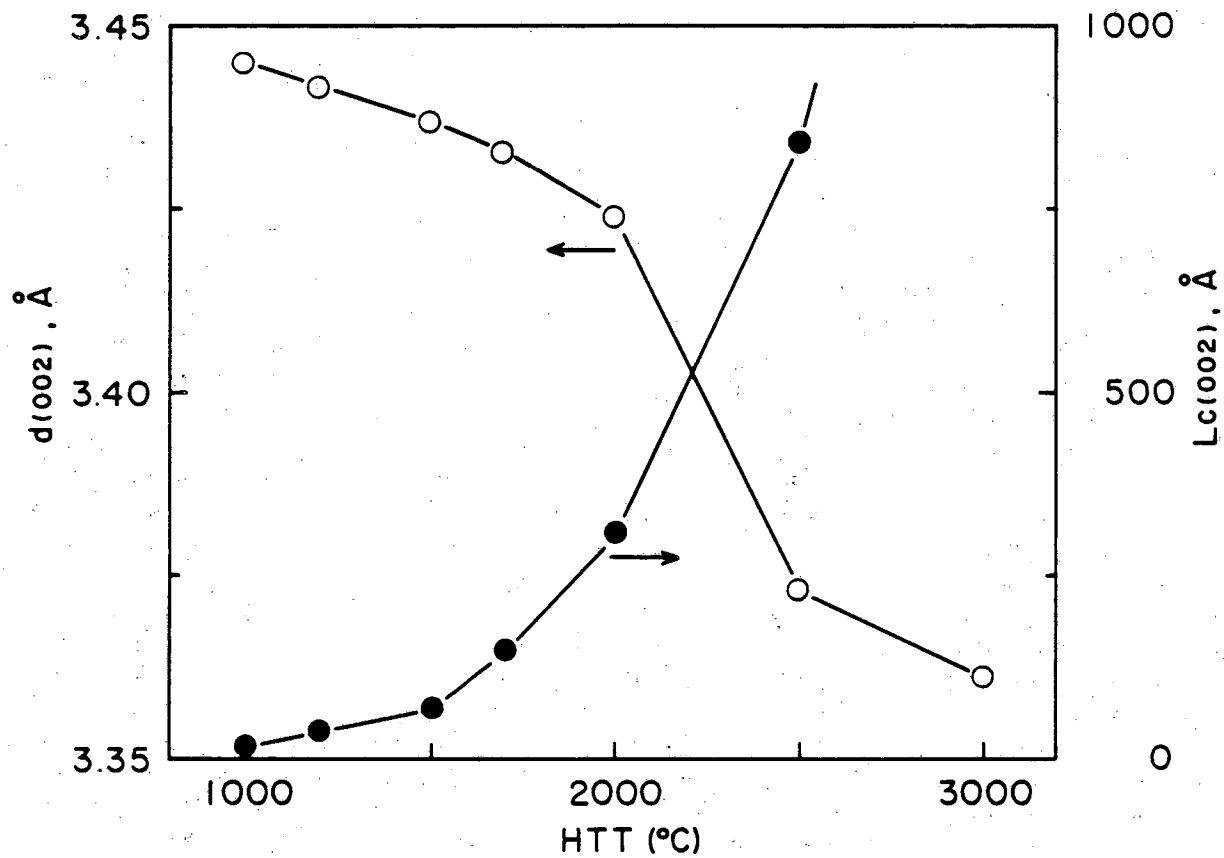
Fig. 5. X-ray diffraction profiles of modified pitch coke.

- (a) Heat-treated at 2000°C
- (b) Heat-treated at 2500°C
- (c) Heat-treated at 3000°C



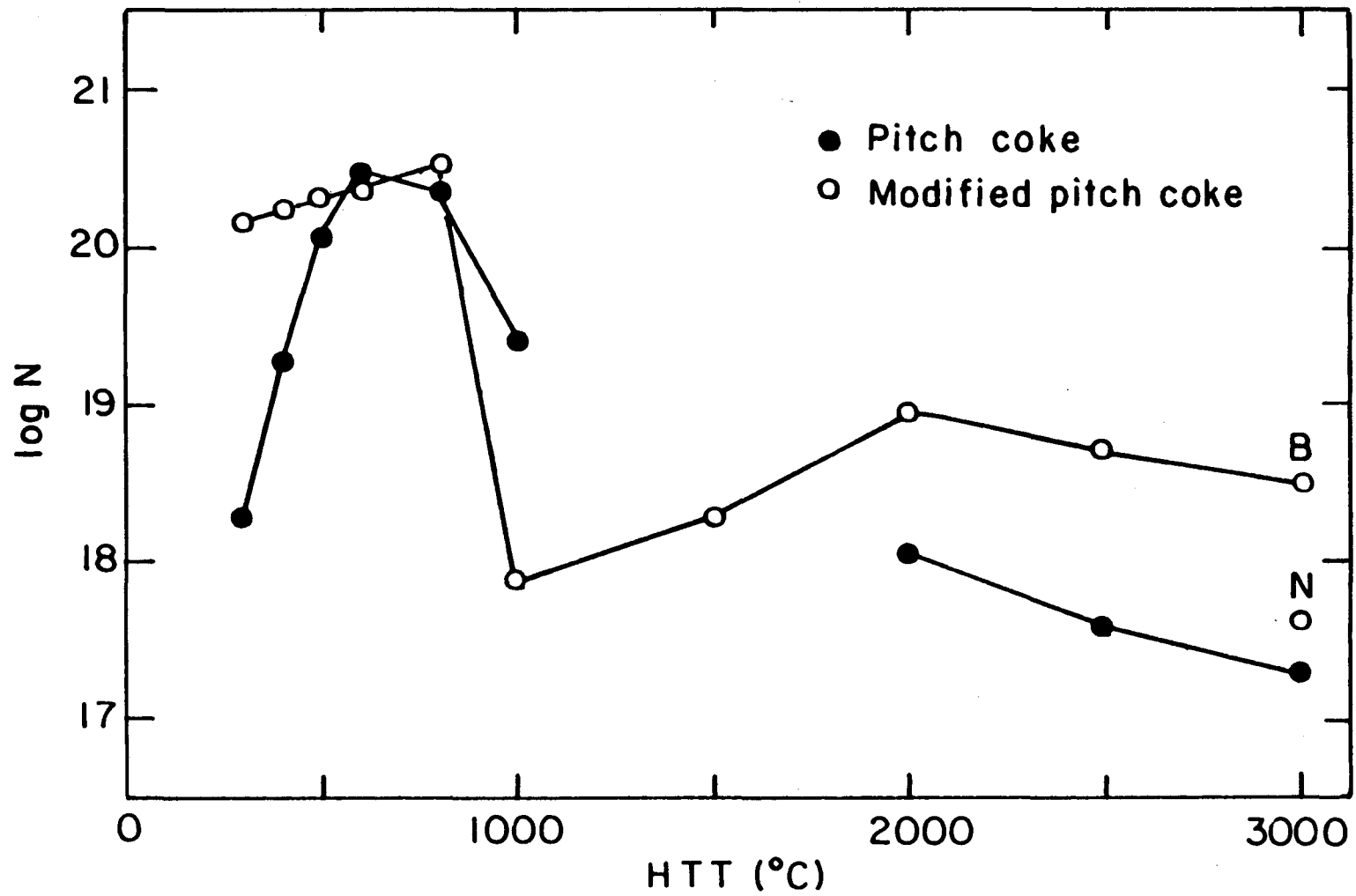
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Fig. 6. Scheme of separation of composite profile shown in Fig. 5(c).



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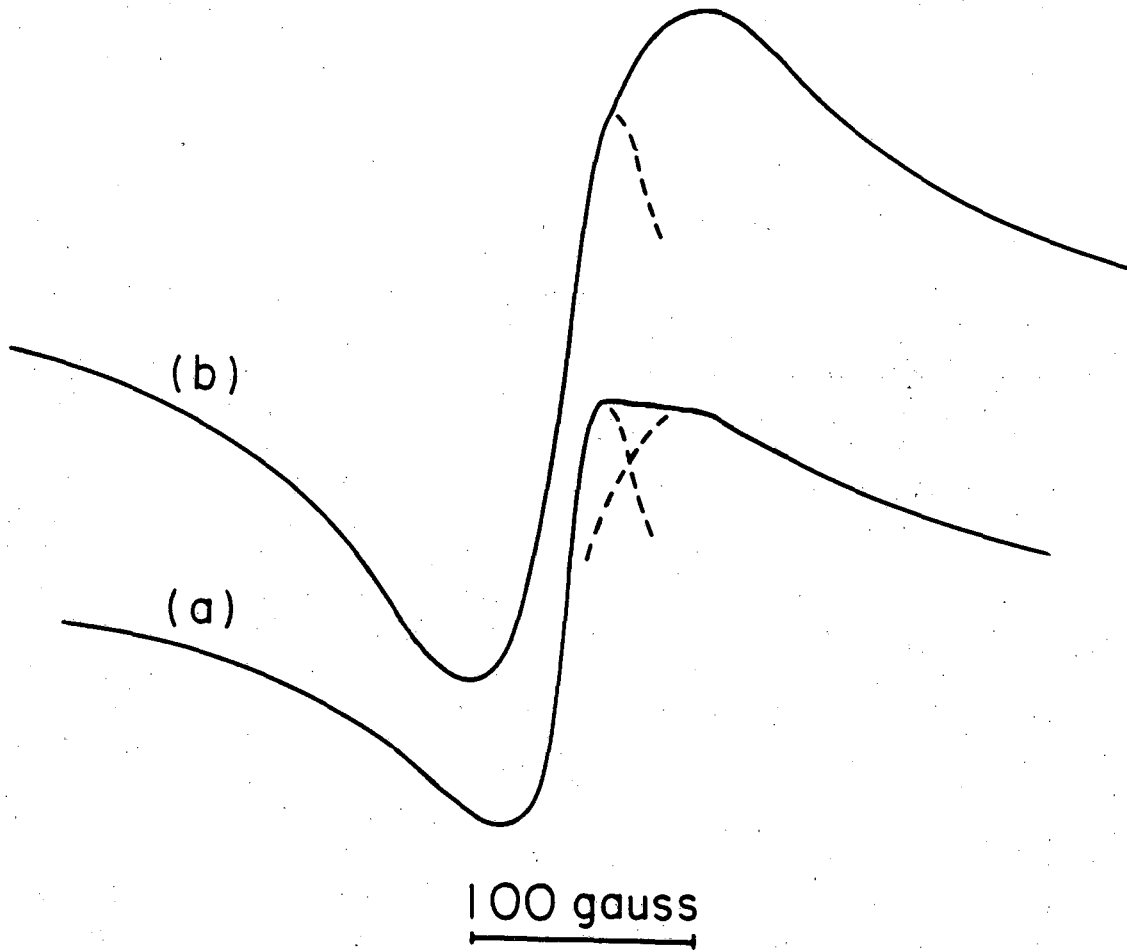
Fig. 7. Changes in interlayer spacing (d_{002}) and crystallite thickness (L_c) of pitch coke.



XBL 8411-6097

Fig. 8. Unpaired electron concentration (N) as a function of HTT.

B: Broad-line component
N: Narrow-line component



XBL8412-5870

Fig. 9. ESR spectrum of modified pitch coke heat-treated at 3000°C.

- (a) Measured at 20°C in vacuum
- (b) Measured at -160°C in vacuum

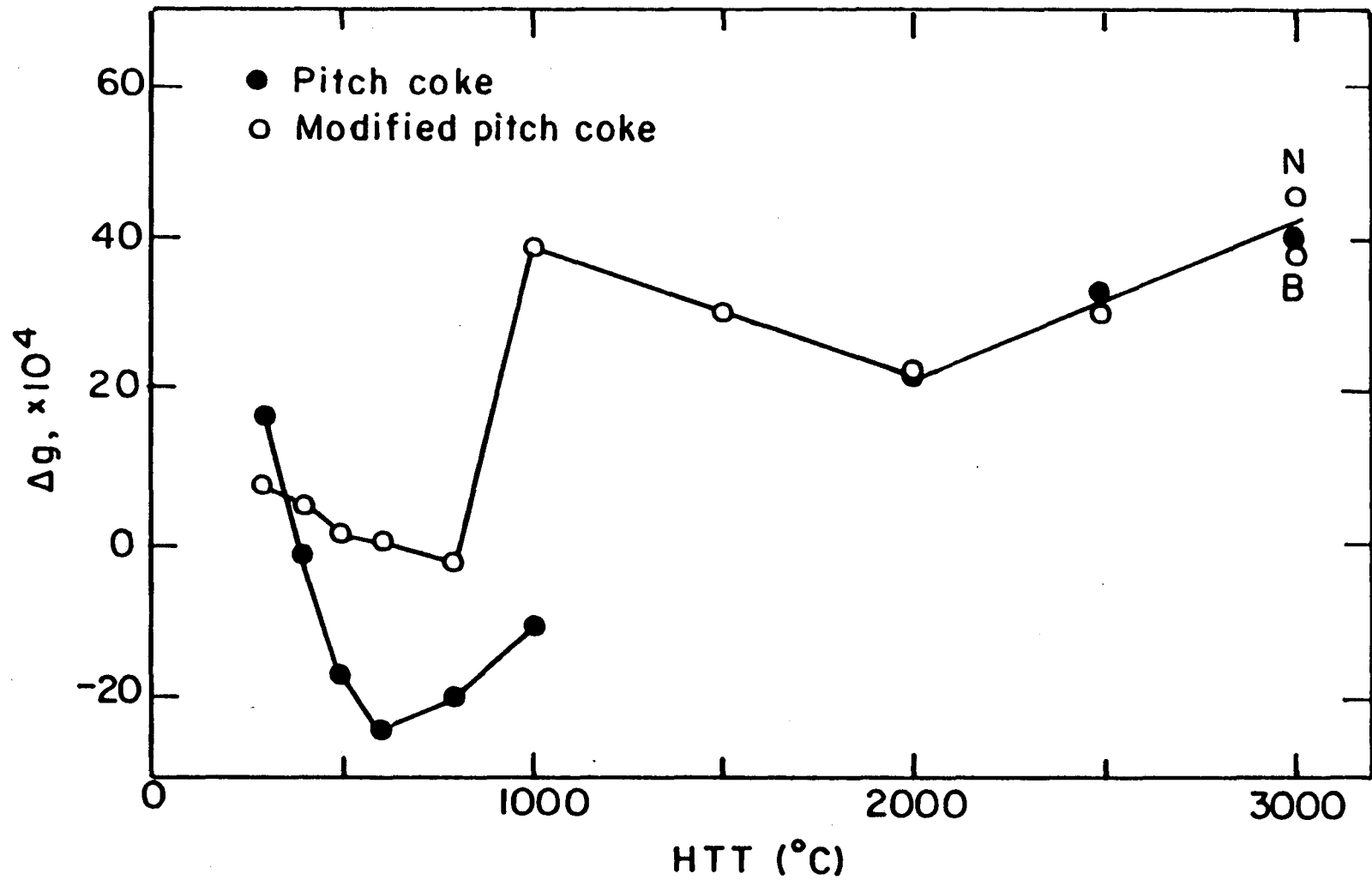
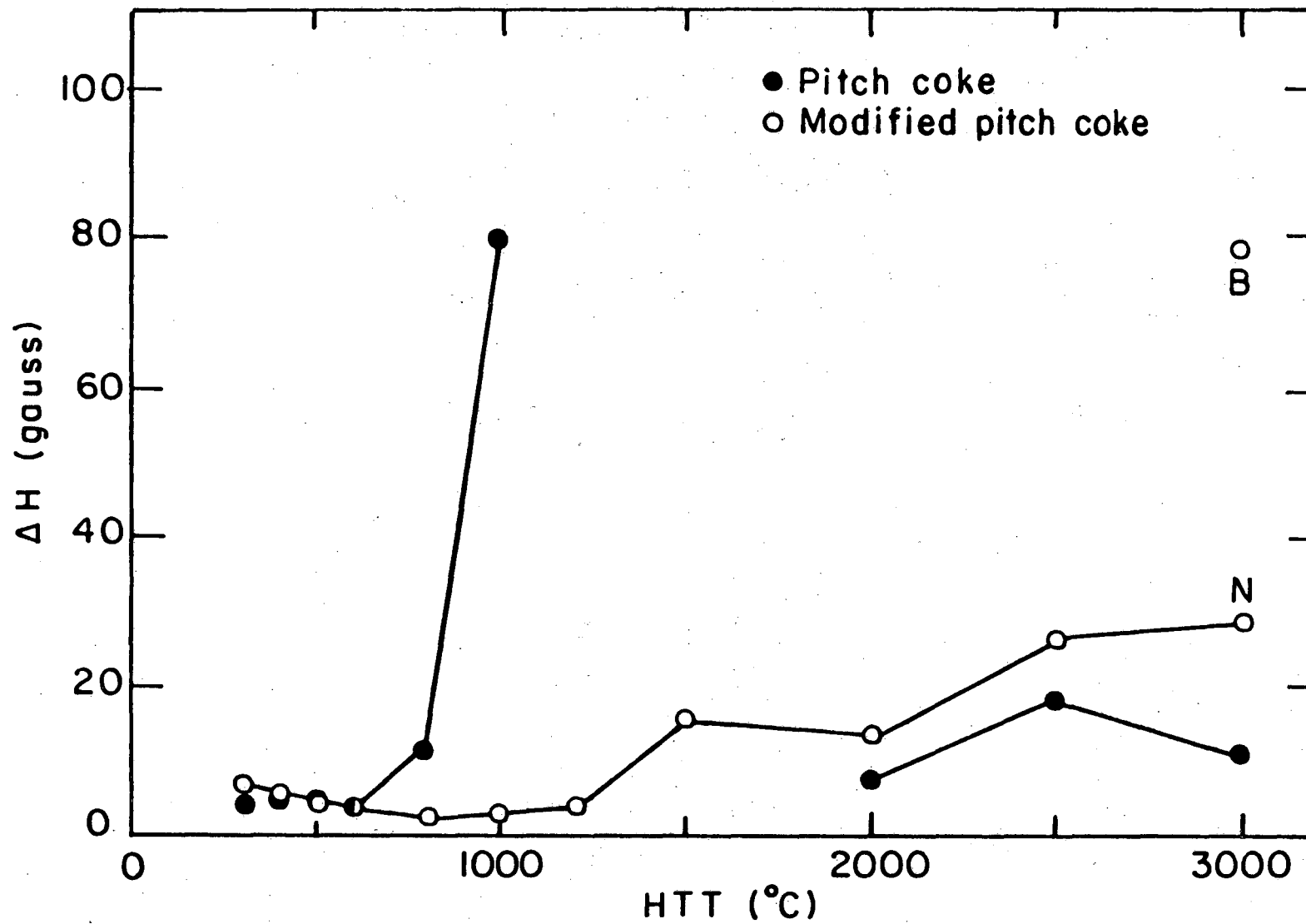


Fig. 10. Changes in Δg with HTT.

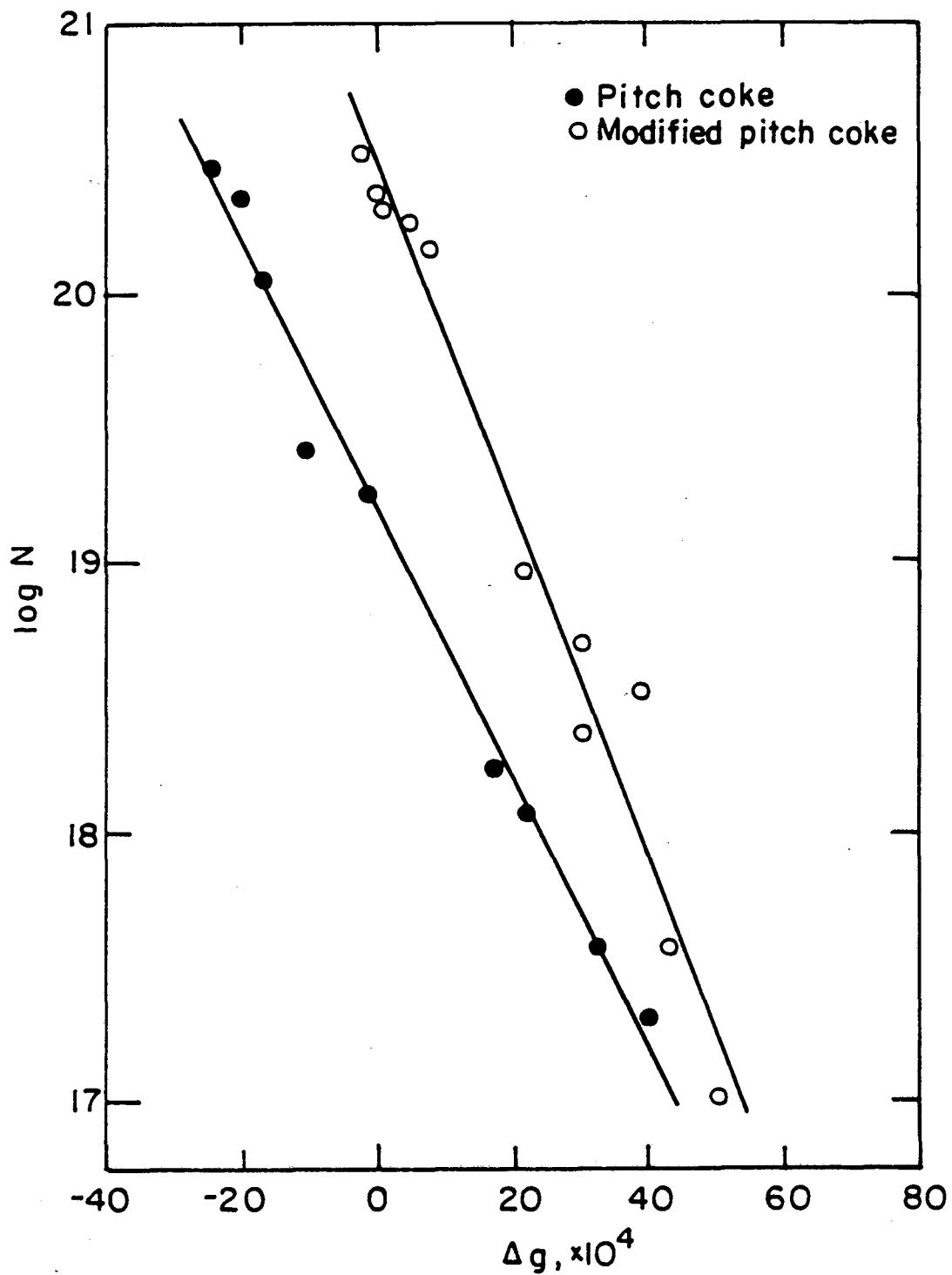
B: Broad-line component
N: Narrow-line component

XBL 8411-6098



XBL 8411-6099

Fig. 11. Changes of line width (ΔH) of ESR spectrum of pitch coke and modified pitch coke with HTT.



XBL 8411-6100

Fig. 12. Relation between unpaired electron concentration (N) and Δg .

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