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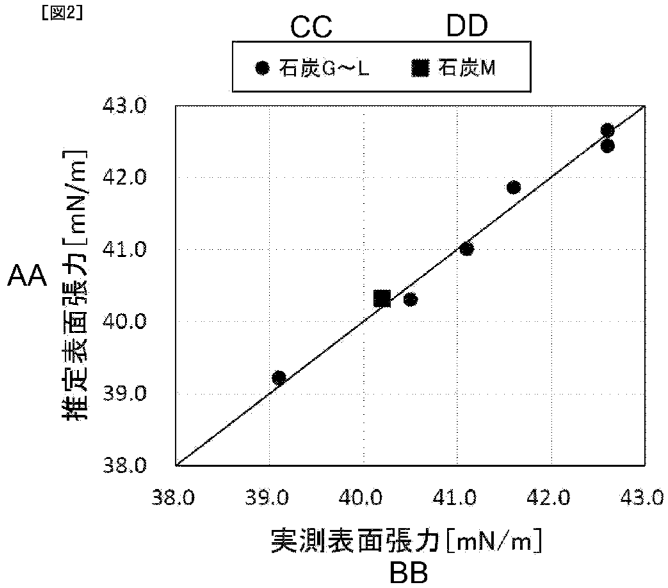
(54) **Title:** METHOD FOR ESTIMATING COAL SURFACE TENSION, AND METHOD FOR PRODUCING COKE

(54) 発明の名称： 石炭の表面張力推定方法およびコークスの製造方法

［図2］



AA Estimated surface tension
BB Actual measured surface tension
CC Coals
DD Coal

(57) **Abstract:** Provided is a method for conveniently estimating coal surface tension. This method for estimating coal surface tension comprises performing multiple regression analysis of the total inert quantity and physical values representing the surface tension and rank of coalification for a plurality of brands of coal to create a regression formula in advance that uses a criterion variable as the coal surface tension and that includes the physical values and the total inert quantity as explanatory variables, measuring the total inert quantity and the physical value of the coal for estimating surface tension, and calculating the coal surface tension using the measured physical values, the total inert quantity, and the regression formula.

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(57) 要約：簡便に石炭の表面張力を推定する方法を提供する。石炭の表面張力推定方法であって、複数の銘柄の石炭における表面張力、石炭化度を表す物性値および全イナート量を重回帰分析し、目的変数を石炭の表面張力とし、説明変数として前記物性値および前記全イナート量を含む回帰式を予め作成しておき、表面張力を推定する石炭の前記物性値と全イナート量とを測定し、測定された前記物性値および前記全イナート量と前記回帰式とを用いて石炭の表面張力を算出する。

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METHOD FOR ESTIMATING SURFACE TENSION OF COAL AND METHOD FOR PRODUCING COKE

Technical Field

[0001]

The present invention relates to a method for estimating the surface tension of coal and a method for producing coke.

Background Art

[0002]

Coke used as a blast furnace raw material for pig-iron production in blast furnaces preferably has high strength. This is because coke having low strength degrades in blast furnaces to inhibit gas permeability in blast furnaces, which hinders stable production of pig iron.

[0003]

Coke is produced by carbonizing coal. Carbonization is a process for heating coal at a pyrolysis temperature or higher (about 300°C or higher) in a non-oxidizing atmosphere. Coal that softens and melts at 350°C to 600°C in a carbonization process is preferably used as a raw material of coke. When softening and melting, coal powder or particles adhere to and fuse with each other to form lump coke.

[0004]

To produce coke having high strength, coal particles preferably adhere well to each other. The surface tension of heat-treated coal (semicoke) is used as a physical property value for evaluating the adhesiveness of the coal.

[0005]

Examples of the method for measuring the surface tension of coal include a capillary-rise method, a maximum bubble pressure method, a drop weight method, a pendant drop method, a ring method, a Wilhelmy method, an advancing/receding contact angle method, a tilting plate method, and a film flotation method. Since coal is composed of various molecular structures and thus expected to have uneven surface tension, the film flotation method in Non Patent Literature 1 or Patent Literature 1 expected to evaluate the surface tension distribution is said to be the most reasonable measurement method.

[0006]

The film flotation method is a technique based on the idea that pulverized sample particles placed in liquid and starting to sink from floating state have the same surface tension as the liquid have. Sample particles are dropped into liquids having various surface tensions, and the mass ratio of sample particles that float in each liquid is determined. The surface tension distribution is obtained from the result. The film flotation method can measure the surface tension of any coal, regardless of the type of coal, such as hard coking coal, non- or slightly caking coal, anthracite, and heat-treated coal (semicoke) made by treating such coal with heat.

Citation List

Patent Literature

[0007]

Patent Literature 1: Japanese Patent No. 5737473

Non Patent Literature

[0008]

Non Patent Literature 1: D. W. Fuerstenau: International Journal of Mineral Processing, 20 (1987), 153

Summary of Invention

[0009]

The film flotation method has a problem of taking a long time (about one day) to measure the surface tension of coal and is not effective in terms of time. The film flotation method also has a problem of a complicated process for measuring the surface tension, and only skilled measurers can stably measure the surface tension. The present invention attempts to deal with these problems that occur in measuring the surface tension of coal and provides a method for easily estimating the surface tension of coal.

[0010]

The present invention provides the following.

(1) A method for estimating a surface tension of coal includes: subjecting a surface tension, a physical property value representing a coal rank, and a total inert content of each of different brands of coal to multiple regression analysis to determine in advance a regression equation including the surface tension of coal as an objective variable and the physical property value and the total inert content as explanatory variables; and measuring the physical property value and the total inert content of a coal of which the surface tension is to be estimated, and calculating the surface tension of the coal by using the measured physical property value and the measured total inert content, and the regression equation, wherein the total inert content (vol%) is calculated in accordance with JIS M 8816 using the following formula:

Inert content (vol%) = fusinite (vol%) + micrinite (vol%) + (2/3) × semifusinite (vol%) + mineral matter (vol%).

(2) In the method for estimating a surface tension of coal according to (1), the physical property value is a mean maximum vitrinite reflectance of the coal.

(3) In the method for estimating a surface tension of coal according to (1) or (2), the surface tension is a surface tension of a semicoke made by heating coal to a temperature of 350°C or higher and 800°C or lower.

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(4) A method for producing coke includes: a step of estimating the surface tensions of coals by the method for estimating a surface tension of coal according to any one of (1) to (3), blending coals having similar surface tensions

estimated by the method for estimating a surface tension of coal according to any one of (1) to (3) to form a coal blend; and carbonizing the coal blend to produce coke.

[0011]

The surface tension of coal can easily be estimated by carrying out the method for estimating a surface tension of coal according to the present invention. When the surface tension of coal can easily be estimated in this way, the estimated value of the surface tension can be used to investigate blending of coals, which enables production of coke with high quality.

Brief Description of Drawings

[0012]

[Fig. 1] Fig. 1 is a graph showing plots (3 points) of the surface tension of samples having different inert contents and the regression line of the plots for each of samples having different inert contents in 6 brands (A to F) of coal.

[Fig. 2] Fig. 2 is a graph showing the relationship between the measured surface tensions and the estimated surface tensions of coals G to M.

[Fig. 3] Fig. 3 is a graph showing plots (3 points) of the surface tension of samples having different inert contents and the regression line of the plots for each of 3 brands (N, O, P) of coal with a heat treatment temperature of 400°C.

[Fig. 4] Fig. 4 is a graph showing plots (3 points) of the surface tension of samples having different inert contents and the regression line of the plots for each of 3 brands (N, O, P) of coal with a heat treatment temperature of 600°C.

Description of Embodiments

[0013]

The present invention will be described below through embodiments of the present invention. The inventors of the present invention focus on coal components that soften and melt with heat (hereinafter referred as reactives) and coal components that neither soften nor melt with heat (hereinafter referred to as inerts). First, the relationship between the surface tensions of the reactives and the inerts and the surface tension of coal will be described.

[0014]

Since coal inerts are harder than reactives, the inerts tend to concentrate in coarse particles of coal after pulverizing. This tendency is used to prepare samples having different inert contents from the same brand of coal by pulverizing and sifting. The total inert content (hereinafter may be referred to as TI) of each of the samples having different inert contents prepared in this way is measured, and the samples are each treated with heat at a predetermined temperature to form semicokes. The TI is the total inert content defined in JIS M 8816 and indicates the proportion (vol%) of inerts contained in coal.

[0015]

In this embodiment, the coal of which the surface tension is to be estimated includes heat-treated coal, that is, semicoke. The method for estimating the surface tension of coal according to this embodiment can be applied to coal without a heat treatment as well as semicoke. Since the surface tension of semicoke is particularly useful for predicting coke strength and producing coke with high strength, the method for measuring the surface tension of semicoke, which is heat-treated coal, will be described in this embodiment. In this embodiment, the semicoke is produced in the following (a) to (c).

(a) Pulverizing coal. With regard to the size of particles of pulverized coal, the coal is preferably pulverized to a particle size of 250 μm or less, more preferably pulverized to 200 μm or less, which is the size of particles in proximate analysis of coal described in JIS M8812, in order to prepare uniform samples from coal which is not uniform in macerals and properties.

(b) Heating the pulverized coal to a temperature of 350°C or higher and 800°C or lower at an appropriate heating rate with no air or in an inert gas. The heating rate is preferably set according to the heating rate during production of coke in a coke oven.

(c) Cooling the heated coal in an inert gas to produce semicoke.

[0016]

With regard to the heating temperature for heating the coal, the coal is preferably heated to a temperature between 350°C at which the coal starts to soften and melt and 800°C at which coking is complete, based on the idea that the surface tension has an effect on adhesion between coal particles. However, in a range of heating temperatures of 350°C to 800°C, the temperature that particularly contributes to adhesion is 350°C to 550°C which is a temperature of softening and melting, and the adhesion structure may be set around 500°C. For this, the heating temperature is particularly preferably 480°C to 520°C, which is around 500°C, and the heating temperature is set to 500°C in this embodiment. Heating is preferably performed in an inert gas (e.g., nitrogen, argon, helium) atmosphere, which is unreactive with coal.

[0017]

Cooling is preferably performed in an inert gas atmosphere, which is unreactive with coal. The heat-treated coal is preferably quenched at a cooling rate of 10 °C/sec or more. The reason for quenching is to maintain a molecular structure in the reactive state, and the cooling rate is preferably 10 °C/sec or higher at which the molecular structure may not change. Quenching may be performed by using liquid nitrogen, iced water, water, or an inert gas, such as a nitrogen gas. Quenching is preferably performed by using liquid nitrogen.

[0018]

The surface tension of the coal can be measured by using the film flotation method described in Non Patent Literature 1. This method can be used for both coal and semicoke made from the coal, and the surface tension distribution can be obtained by using a finely pulverized sample. The mean of the obtained surface tension distribution is defined as a representative value of the surface tension of the sample. The measurement of the surface tension of semicoke using the film flotation method is specifically described in Patent Literature 1.

[0019]

Fig. 1 is a graph showing plots (3 points) of the surface tension (mean of surface tension distribution) of samples having different inert contents and the regression line of the plots for each of 6 brands (A to F) of coal treated with heat at 500°C (semicokes). In Fig. 1, the horizontal axis represents TI (%), and the vertical axis represents the surface tension (mN/m). Each regression line is a simple regression equation of the surface tension against TI and calculated by using the least squares method so as to minimize the error between the simple regression equation and each plot. As shown in Fig. 1, an approximately linear relationship is observed between TI and surface tension for each brand of coal. A value corresponding to TI = 100 on the regression line is an estimated value of the surface tension at 100% inerts (hereinafter may be referred to as γ_{100}), and a value corresponding to TI = 0 is an estimated value of the surface tension at 100% reactives (hereinafter may be referred to as γ_0).

[0020]

Fig. 1 shows that γ_0 has a tendency of convergence to a substantially constant value, regardless of the brand of coal, and γ_{100} does not have a tendency of convergence and greatly varies according to the brand of coal. Since a linear relationship is observed between surface tension and TI, and γ_{100} greatly varies according to the brand of coal, the TI and γ_{100} are considered as dominant factors that have an effect on the surface tension of coal.

[0021]

The inventors of the present invention have studied the relationship between γ_{100} and coal properties and have found that γ_{100} shows a strong correlation with the mean maximum vitrinite reflectance (hereinafter may be referred to as R_O) of coal. With TI and R_O as main dominant factors that have an effect on the surface tension of coal, it is determined whether the surface tension of coal can be estimated from the measured values of TI and R_O. Table 1 shows the properties of the coals G to M, which are used in the determination. R_O is an example physical property value representing a coal rank. Examples of physical property values representing coal ranks other than R_O include the volatile matter of coal, the carbon content, and the re-solidification temperature in softening and melting. These physical property values all show a good correlation with R_o. Thus, the volatile matter of coal, the carbon content, or the re-solidification temperature in softening and melting can thus be used as a dominant factor that has an effect on the surface tension, instead of R_O. These physical property values can be used as an explanatory variable in multiple regression analysis described below.

[0022]

[Table 1]

Brand	logMF	R_o	TI	Measured Surface Tension	Estimated Surface Tension
	log/ddpm	%	%	mN/m	mN/m
G	2.97	1.20	20.36	40.5	40.3
H	0.48	1.56	20.96	39.1	39.2
I	2.94	0.97	33.98	41.6	41.9
J	2.78	0.98	47.39	42.6	42.7
K	2.77	0.97	43.40	42.6	42.4
L	1.34	1.30	36.88	41.1	41.0
M	1.67	1.23	22.10	40.2	40.3

[0023]

In Table 1, "logMF (log/ddpm)" is a common logarithmic value of the maximum fluidity (MF/ddpm) of coals measured by the Gieseler plastometer method described in JIS M8801. "R_o (%)" is a mean maximum vitrinite reflectance of coals G to M in JIS M 8816. "TI (%)" is a total inert content (vol%) and calculated in accordance with Methods of microscopical measurement for the macerals for coal and coal blend in JIS M 8816 and the following formula (1) based on the Parr formula described in explanation of the Methods.

[0024]

Inert content (vol%) = fusinite (vol%) + micrinite (vol%) + (2/3) × semifusinite (vol%) + mineral matter (vol%) ··· (1)

The "measured surface tension (mN/m)" is a surface tension (representative value) obtained by measuring semicokes made by treating coals G to M with heat at 500°C in accordance with the film flotation method. The "estimated surface tension (mN/m)" is an estimated surface tension calculated by using the measured values of R_O and TI and the regression equation including the surface tension (Y) as an objective variable and R_O and TI as explanatory variables (X_1, X_2).

[0025]

The coals in Table 1 are examples of coal commonly used as a coke raw material. Coal used as a coke raw material has an MF of 0 to 60000 ddpm (log MF: 4.8 or less), a R_O of 0.6% to 1.8%, and a TI of 3 to 50 vol%. The method for estimating the surface tension of coal according to this embodiment can be suitably used for coals in such ranges.

[0026]

The regression equation including the surface tension as an objective variable and R_O and TI as explanatory variables can be represented by the following formula (2).

[0027]

$$\text{Surface tension} = a + b_1 \times R_O + b_2 \times TI \quad \cdots (2)$$

In the formula (2), a, b_1, and b_2 are parameters of the regression equation.

[0028]

In this embodiment, the measured surface tensions and the measured values of R_O and TI of different brands of coals G to L are subjected to multiple regression analysis to calculate the parameters of the formula (2) and thus to obtain the following regression equation (3).

[0029]

Estimated surface tension = 42.805 - 3.123R_O + 0.0614TI ··· (3)

In Table 1, the "estimated surface tension (mN/m)" is an estimated surface tension calculated by using the regression equation (3). Coal M is not used to calculate the parameters of the regression equation (3), but the estimated surface tension of coal M calculated by using the regression equation (3) is substantially the same as the measured surface tension of coal M.

[0030]

Fig. 2 is a graph showing the relationship between the measured surface tensions and the estimated surface tensions of coals G to M. In Fig. 2, the horizontal axis represents the measured surface tension (mN/m), and the vertical axis represents the estimated surface tension (mN/m). In Fig. 2, the solid circle plots represent coals G to L in Table 1, and the solid square plots represent coal M in Table 1. Fig. 2 indicates a very strong correlation between the measured surface tensions and the estimated surface tensions. This result demonstrates that the surface tension of coal can be accurately estimated by using the method for estimating the surface tension of coal according to this embodiment.

[0031]

Fig. 2 shows an example of estimating the surface tension of coals treated with heat at 500°C, but the heat treatment temperature of coals in this embodiment is not limited to 500°C. To confirm that the method for estimating the surface tension of coal according to this embodiment is not limited to the case of heat treatment at 500°C, it is determined whether the relationship between TI and surface tension shown in Fig. 1 is also established at other heat treatment temperatures.

[0032]

Samples having different TI contents are prepared by the above method using 3 brands (N, O, P) of coal. The samples are converted into semicokes according to the method including (a) to (c) described above under the same conditions except that only the heat treatment temperature is changed to 400°C and 600°C. The surface tension of each semicoke is measured, and the relationship between TI and surface tension is determined in the same manner as in Fig. 1.

[0033]

Fig. 3 is a graph showing plots (3 points) of the surface tension of samples having different inert contents and the regression line of the plots for each of 3 brands (N, O, P) of coal with a heat treatment temperature of 400°C. Fig. 4 is a graph showing plots (3 points) of the surface tension of samples having different inert contents and the regression line of the plots for each of 3 brands (N, O, P) of coal with a heat treatment temperature of 600°C. In Fig. 3 and Fig. 4, the horizontal axis represents TI (%), and the vertical axis represents the surface tension (mN/m).

[0034]

As shown in Fig. 3 and Fig. 4, a relationship similar to that in Fig. 1 is established between the TI and the surface tension of semicokes prepared at different heat treatment temperatures, and this tendency does not change for the same coal. Since a relationship similar to that in Fig. 1 is established between the TI and the surface tension even when the heat treatment temperature is changed, the method for estimating the surface tension of coal according to this embodiment can be used for semicokes prepared at different temperatures.

[0035]

Patent Literature 1 also discloses that the surface tensions of semicokes prepared at heat treatment temperatures of 350°C or higher and 800°C or lower show the same tendency regardless of the type of coal. This indicates that the method for estimating the surface tension of coal according to this embodiment can be used for semicokes prepared at a temperature of 350°C or higher and 800°C or lower as well as semicokes made by a heat treatment at 500°C. In other words, estimation of the surface tension of a coal treated with heat at a predetermined temperature of 350°C or higher and 800°C or lower can be done by using the regression equation obtained by multiple regression analysis using the data of surface tensions obtained by treating coals at the predetermined temperature.

[0036]

In general, coal maceral analysis regarding TI, physical property values representing coal ranks, such as R_o, and other parameters are widely used in business transactions for the purpose of expressing the characteristics of coal, and these parameters are analyzed and available. Therefore, as long as the surface tension of a coal can be estimated from the coal rank and the TI of the coal, the surface tension of the coal can be estimated without relying on skilled measurers, and the time for measuring the surface tension can be saved.

[0037]

When the regression equation (3) is determined in advance, the measurement of R_O and TI of a coal of which the surface tension is to be estimated allows estimation of the surface tension of the coal. The surface tension of the coal can thus be estimated accurately, easily, and readily by carrying out the method for estimating the surface tension of coal according to this embodiment. The strength of a coke made from a coal blend containing a mixture of coals with different surface tensions is lower than that of a coke made from a coal blend containing a mixture of coals with similar surface tensions. If the surface tension of coal can be estimated in this way, the estimated value of the surface tension can be used to investigate blending of coals. The use of a coal blend having the blending ratio set by the blending investigation to produce coke thus enables production of coke with high quality.

[0038]

The reference in this specification to any prior publication (or information derived from it), or to any matter which is known, is not, and should not be taken as an acknowledgment or admission or any form of suggestion that that prior publication (or information derived from it) or known matter forms part of the common general knowledge in the field of endeavour to which this specification relates.

[0039]

Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" and "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

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THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

[Claim 1]

A method for estimating a surface tension of coal, the method comprising:

subjecting a surface tension, a physical property value representing a coal rank, and a total inert content of each of different brands of coal to multiple regression analysis to determine in advance a regression equation including the surface tension of coal as an objective variable and the physical property value and the total inert content as explanatory variables; and

measuring the physical property value and the total inert content of a coal of which the surface tension is to be estimated, and calculating the surface tension of the coal by using the measured physical property value and the measured total inert content, and the regression equation

wherein the total inert content (vol%) is calculated in accordance with JIS M 8816 using the following formula:

Inert content (vol%) = fusinite (vol%) + micrinite (vol%) + (2/3) × semifusinite (vol%) + mineral matter (vol%).

[Claim 2]

The method for estimating a surface tension of coal according to Claim 1, wherein the physical property value is a mean maximum vitrinite reflectance of the coal.

[Claim 3]

The method for estimating a surface tension of coal according to Claim 1 or 2, wherein the surface tension is a surface tension of a semicoke made by heating coal to a temperature of 350°C or higher and 800°C or lower.

[Claim 4]

A method for producing coke, the method comprising:

a step of estimating the surface tensions of coals by the method for estimating a surface tension of coal according to any one of claims 1 to 3,

blending coals having similar surface tensions estimated by the method for estimating a surface tension of coal according to any one of Claims 1 to 3 to form a coal blend; and

carbonizing the coal blend to produce coke.

FIG. 1

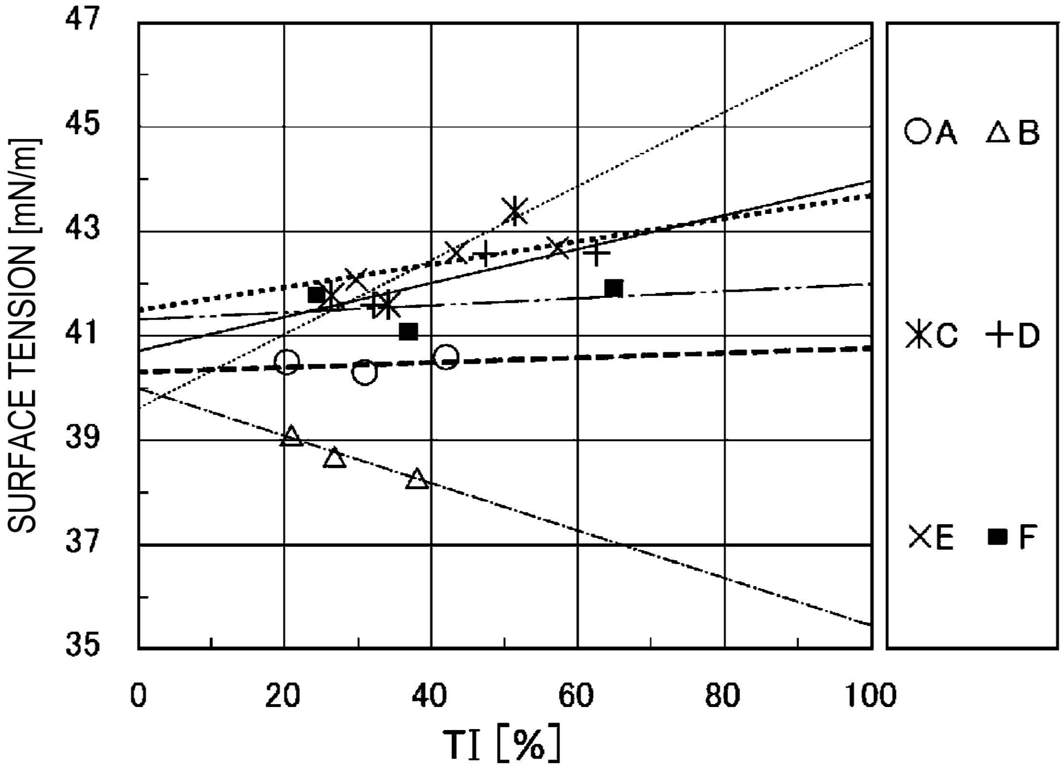

47
45
43
41
39
37
35
SURFACE TENSION [mN/m]
0
20
40
60
80
100
TI [%]
A
B
C
D
E
F

FIG. 2

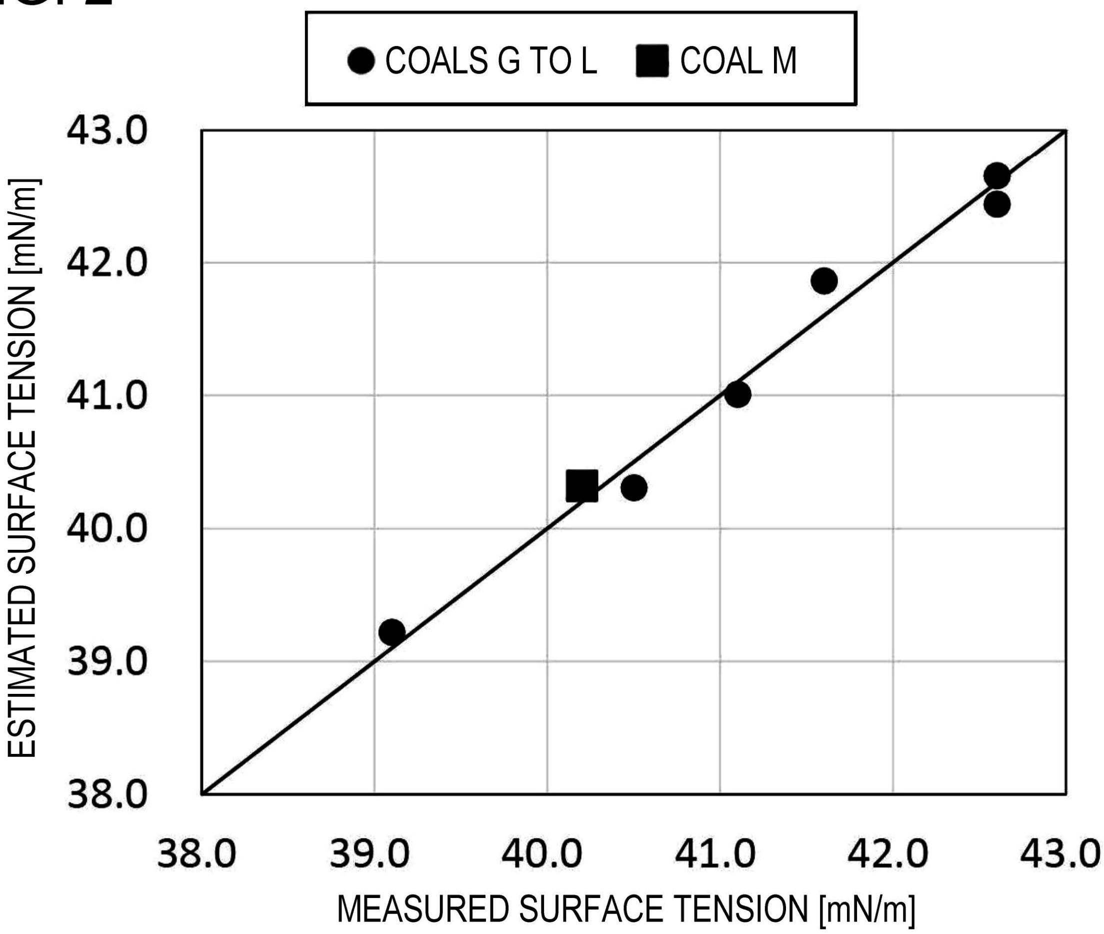

COALS G TO L
COAL M
43.0
42.0
41.0
40.0
39.0
38.0
ESTIMATED SURFACE TENSION [mN/m]
38.0
39.0
40.0
41.0
42.0
43.0
MEASURED SURFACE TENSION [mN/m]

FIG. 3

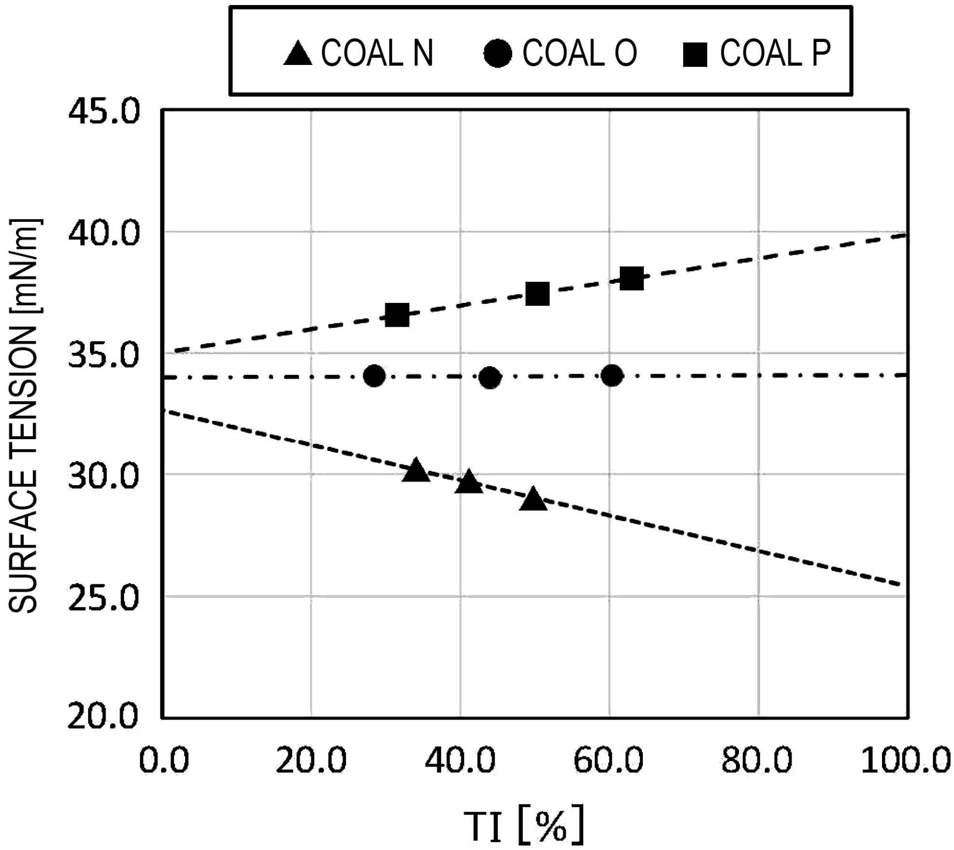
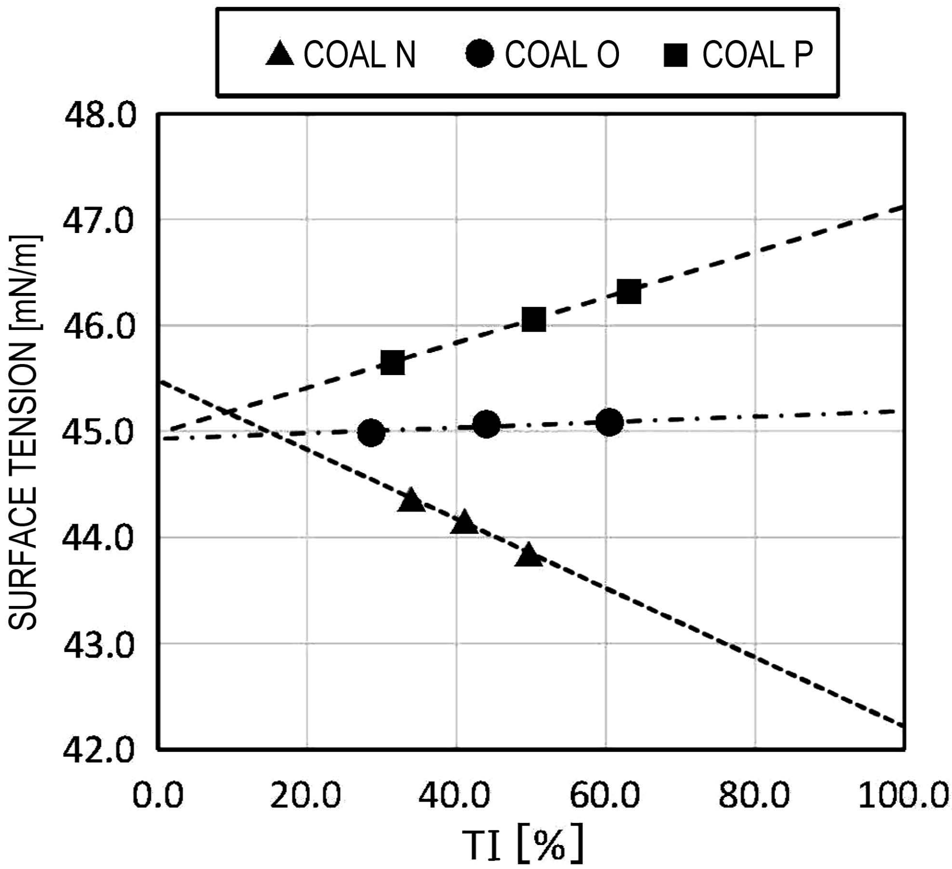

COAL N
COAL O
COAL P
SURFACE TENSION [mN/m]
45.0
40.0
35.0
30.0
25.0
20.0
0.0
20.0
40.0
60.0
80.0
100.0
TI [%]

FIG. 4


COAL N
COAL O
COAL P
SURFACE TENSION [mN/m]
48.0
47.0
46.0
45.0
44.0
43.0
42.0
0.0
20.0
40.0
60.0
80.0
100.0
TI [%]