A Method for Generating High Pressure by Restraining Thermal Expansion*

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The high-pressure apparatus for research and production usually uses a pressure produced by an external force. Such apparatus cannot be prevented from pressure-leakage and frictional force. In this paper, a new type of apparatus is devised which generates a high pressure by converting thermal energy to mechanical one in a thermally isolated system. The energy conversion is done by restraining the thermal expansion of the system. The pressure-leakage and frictional force do not appear in the new type apparatus. The pressure generated in this apparatus is investigated by the theoretical calculation and experiment. A method for measuring the pressure under the high temperature is established in the process of experiment. As the result of investigation, it is ascertained that a high pressure can be obtained by this apparatus and that the theoretical values agree well with the experimental values.

Key Words : Thermal Conduction, High Pressure Generation, Induction Heating, Thermal Stress, Pressure Measurement, Transformation, Resistivity Measurement, Insulating Film, Dual Difference Method, Finite Element Analysis

1. Introduction

The high-pressure apparatus by mechanical pressing method is classified into four types according to the method of pressing: piston-cylinder type,⁽¹⁾⁽²⁾ Bridgman-anvil type,⁽³⁾⁽⁴⁾ belt type⁽⁵⁾⁽⁶⁾ and multi-anvil type.⁽⁷⁾ The belt-type is the commonest apparatus for the synthesis of diamond, in which the normal pressure is under 10 GPa. The mechanical type of high-pressure apparatus cannot be prevented from pressure-leakage and frictional force. Proper means of solving this problem have not been found yet. High temperature and high pressure can be obtained by generating thermal energy in a thermally isolated system which can be kept at a constant volume. In this study, a system on this concept is constructed of a material with great thermal expansion coefficient and great bulkmodulus enormously expanding on melting, which is surrounded by an insulator with great Young's modulus. Then the possibility of the method of generating a high tempera-

ture and a high pressure by supplying thermal energy to the above system is investigated by theoretical calculations and experiments. It is obvious from the structure of the system that the problem of pressure-leakage and frictional force is solved by this method.

In order to investigate the possibility of this method theoretically and experimentally, methods for calculating the generated pressure and measuring the high pressure under high temperature are proposed. Subsequently, a high pressure apparatus composed of a cemented carbide ball surrounded by a sintered alumina is made on an experimental basis. As the result of calculating and measuring the pressure generated in this apparatus, it was ascertained from the measurements that the high pressure estimated by the calculation could be generated.

2. Numerical Calculation of Pressure Generated Restraining Thermal Expansion

The preceding system of generating a high temperature and a high pressure can be embodied by a high-pressure apparatus which generates a high temperature and a high pressure by means of supplying thermal energy to a good conductor ball located at the center of the insulator. The shape of the conductor was chosen to

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be a sphere by considering the strength of the insulator and the effect of heat insulating the conductor. The shape of the insulator was chosen to be a cylinder to avoid the difficulty of production. The larger the external dimensions of insulator cylinder, the better to avoid a break in the insulator. But the efficiency of heating the conductor ball by the induction coil lowers because the diameter of induction coil becomes larger as the dimensions of insulator become larger. Accordingly, the dimensions of insulator were decided in such a way that the tensile stress in the insulator could be smaller than the tensile strength of the insulator.

In order to investigate the generated pressure in the above-mentioned high-pressure apparatus theoretically, the generated pressure must be calculated. In this chapter, the method of calculating the pressure and the temperature generated in the interface between the conductor ball and the insulator is described. First, the temperature distribution is calculated using the dual difference method⁽⁸⁾ from the distribution of electromagnetic energy absorbed in the conductor ball. Secondly, the distribution of thermal stress is calculated using finite element analysis. In the process of the above-mentioned calculation, it was assumed that the electromagnetic energy dissipated in the insulating cylinder was negligibly small.

2.1 Distribution of electromagnetic energy absorbed in a conductor ball.

It is assumed that the electromagnetic energy dissipated in the insulator is so small as to be neglected and that the electric conductivity of conductor ball is homogeneous and depends on the average temperature of it. Thereby, in the same manner of calculating the distribution of electromagnetic energy absorbed in a conductor ball in the air or in a vacuum, the distribution of electromagnetic energy q [J/(m³·s)] absorbed in the conductor ball with radius a in the center of induction coil with radius b shown in Fig. 1 can be calculated using Fomin's solution⁽⁹⁾ and given by equation (1).

$$q = \frac{\pi^2 \omega I_0^2}{c^2 a^2} \left| \sum_{l=1, l: \text{odd}}^{\infty} (-1)^{(l-1)/2} \frac{2l+1}{l(l+1)} \times \frac{l!!}{(l-1)!!} \left(\frac{a}{b} \right)^{2l} k_s a \frac{j_l(k_s a)}{j_{l-1}(k_s a)} P_l(\cos \theta) \right|^2 \quad \dots\dots\dots (1)$$

where I_0 is the amplitude of coil current, $k_s = -(i+1)\sqrt{2\pi\sigma\omega}/c$, c : velocity of electromagnetic wave

in a vacuum, σ : electric conductivity, μ : magnetic permeability, ω : frequency of field.

But it is difficult to measure the amplitude of coil current I_0 directly. Therefore, if the total electromagnetic energy Q absorbed in a conductor ball per unit time can be measured, the amplitude of coil current I_0 can be calculated from the relation between I_0 and Q which is given by equation (2)⁽⁸⁾.

$$Q = \frac{2\pi I_0^2}{\sigma a} \text{Real} \left[i \sum_{l=1, l: \text{odd}}^{\infty} \frac{2l+1}{l(l+1)} \times \left\{ \frac{l!!}{(l-1)!!} \right\}^2 \left(\frac{a}{b} \right)^{2l} k_s a \frac{j_l(k_s a)}{j_{l-1}(k_s a)} \left| \frac{j_l(k_s a)}{j_{l-1}(k_s a)} \right|^2 \right] \quad \dots\dots\dots (2)$$

It can be considered that the total energy absorbed in the conductor ball is quantified by measuring its calorific value. The relation between the total electromagnetic energy and the induction heating time is obtained by measuring the calorific value of the conductor ball heated in various periods by the method of mixtures. Q can be calculated by means of differentiating the equation that expresses the above relation with respect to time.

2.2 Temperature distribution

The temperature distribution of the conductor ball and the insulator cylinder is calculated using the dual difference method from the distribution of electromagnetic energy absorbed in the conductor ball obtained in Chapter 2.1. Since the distribution of electromagnetic energy absorbed in a sphere is symmetric with respect to the axis of cylinder, the calculation of temperature distribution can be treated as a 2-dimensional problem. In the dual difference method, an obtuse-angle triangle can be used as element according to demand, and the centroid of triangle can be chosen as node. Accordingly if the section

Fig. 1 Section of high-pressure apparatus

containing an axis of the cylinder is divided into triangular elements considering the distribution of electromagnetic energy absorption, the number of elements can be decreased without sacrificing numerical accuracy. A dual difference equation is set up by the energy balance of heat conduction across the boundary of element and energy dissipation in an element. The energy dissipation in an element is obtained by means of numerically integrating the electromagnetic energy absorbed in an element.

The heat transfer $\bar{q}$ across the boundary between the conductor ball and the insulator is obtained from the continuity of heat flow and the temperature gradient in the neighborhood of boundary, which is calculated from the temperature in the boundary and the node temperatures of elements adjacent to the boundary. In Fig. 2, let the temperature at the middle point M of line AB which represents a part of the boundary be T_M , and the temperature of the nodes D_1 and D'_1 of elements G_0 and G_1 be T_{D_1} and T'_{D_1} respectively. Then the heat transfer $\bar{q}_{G_0-G_1}$ from element G_0 to element G_1 can be obtained by equation (3).

$$\bar{q}_{G_0-G_1} = k_C \cdot S_{AB} \cdot \frac{T_{D_1} - T_M}{|D_1 M|} = k_I \cdot S_{AB} \cdot \frac{T_M - T'_{D_1}}{|D'_1 M|} \quad \dots\dots\dots (3)$$

Where k_C : thermal conductivity of conductor, k_I : thermal conductivity of insulator, S_{AB} : area of the boundary represented by line AB.

$\bar{q}_{G_0-G_1}$ is given by equation (4) eliminating the boundary temperature T_M from the above equation (3).

$$\bar{q}_{G_0-G_1} = k_C \cdot k_I \cdot S_{AB} \frac{T_{D_1} - T_{D'_1}}{k_C |D_1 M| + k_I |D'_1 M|} \quad \dots\dots\dots (4)$$

Furthermore, it was assumed that the lateral face of cylinder was adiabatic.

Fig. 2 Example for division of the region in the neighborhood of boundary between the conductor ball and the insulator

2.3 Thermal stress distribution

The thermal stress distribution can be calculated by axisymmetric finite element method, because the temperature distribution obtained from the axisymmetric distribution of electromagnetic energy absorption is axisymmetric. The elements for division were the same as those used for the calculation of the temperature distribution. The temperatures of elements for the calculation of thermal stress were the node temperatures calculated in the foregoing paragraph. Furthermore, the boundary condition for the calculation is that both ends of the cylinder are restrained in the direction of axis and that the proper contact and adhesion between the conductor ball and the insulator are ensured.

Since the distribution of electromagnetic energy absorption, temperature distribution and thermal stress can be calculated in the manner described above, the thermal stress generated at each temperature in the process of heating can be calculated according to the calculating order of a flow-chart illustrated in Fig. 3.

2.4 Example

The physical properties and dimensions of a cemented carbide ball and an alumina cylinder are indicated in tables 1 and 2 respectively. In the case of using an induction coil with an inner diame-

Table 1 Physical properties of cemented carbide and alumina

	Cemented carbide	Alumina
Resistivity Ωm	$0.2113 + 9.78 \times 10^{-4}(T-273)^*$	1.05×10^{-2} (12) $\times \exp(-1.21 \times 10^4/T)$
Specific gravity $\times 10^3 \text{ kg/m}^3$	14.0 *	3.92 *
Specific heat J/g·K	$9.77 \times 10^{-5}(T-290) + 0.246$ (10) $(290 \leq T \leq 925)$ $1.44 \times 10^{-5}(T-925) + 0.309$ $(925 \leq T \leq 1300)$	$1.128 + 1.158 \times 10^{-4} T$ (13) $- 3.440 \times 10^{-6} T^2$
Thermal diffusivity m^2/s	$2.38 \times 10^{-5} \exp(-1.2 \times 10^3(T-273))$ *	$9.60 \times 10^{-7} \exp(594/T)$
Coefficient of thermal expansion K^{-1}	6.0×10^{-6} (11)	$3.435 \times 10^{-6}(T-273)^{0.127}$ *
Young's modulus GPa	560 (11)	$400.3 - 0.05048 T$ (14)
Poisson's ratio	0.22 (11)	$0.229 + 1.321 \times 10^{-5} T$ (14)

* measured values

Table 2 Dimensions of high-pressure apparatus used in an experiment

a	1.6 mm
d	10 mm
L_0	12 mm
L_1	5 mm
b	7 mm

ter of 11 mm and 170kHz as a frequency of the magnetic field, the generated pressure was calculated according to the calculating order of the flow-chart illustrated in Fig. 3.

The distribution of dimensionless electromagnetic energy $q/(Q/V)$ absorbed in the cemented carbide ball at room temperature is obtained using equations (1) and (2), which are illustrated in Fig. 4. Considering this distribution, the section containing an axis of the cemented carbide ball and the alumina cylinder was divided into triangular elements as in Fig. 5.

Fig. 6 shows the relationship between the total electromagnetic energy absorbed in the cemented carbide ball $\bar{Q}(J)$ and the induction heating time, which is necessary for calculating the electromagnetic energy absorption at temperatures above room temperature. Thereby the total electromagnetic energy absorption per unit time $\bar{Q}(J/S)$ can be obtained, which is given by equation (5).

$$\left. \begin{aligned} Q &= 25 & (0 \leq \bar{Q} \leq 40) \\ &= 40.27 - 0.382 \times \bar{Q} & (\bar{Q} > 40) \end{aligned} \right\} \dots\dots (5)$$

In the manner described above, the total electromagnetic energy adsorption of the cemented carbide ball per unit time corresponding to its average temperature can be given on the basis of the experimental data. Then the electromagnetic energy absorption can be obtained using equations (1) and (2) according to the flow-chart illustrated in Fig. 3. Solving the dual difference equa-

tions which represent the energy balance of heat conduction across the boundary of elements and energy dissipation in elements, the temperatures of elements are calculated. In the above-mentioned calculation, $T_M(i)$ and $t(j)$ in the flow-chart (see Fig. 2) are equal to $10^\circ\text{C} \times i + \text{room temperature}$ and $1s \times j$ respectively. As an example the temperature distribu-

Fig. 4 Distribution of nondimensional electromagnetic energy absorbed in a cemented carbide ball

Fig. 5 Division of the axial section of cemented carbide ball and alumina cylinder

Fig. 3 Flow-chart for the calculation of generated pressure

Fig. 6 Relationship between the total electromagnetic energy absorbed in a cemented carbide ball and induction heating time

tion of the axial section of cemented carbide ball and alumina in the heating period of 15 s and 50 s is illustrated in Fig. 7 (a) and (b) respectively. Furthermore, the change of temperature distribution with time on the surface of cemented carbide ball is illustrated in Fig. 8. As an example the distribution of σ_r , σ_θ and σ_z in the heating period of 15 s and 50 s is illustrated in Fig. 9 and Fig. 10 respectively. The change of distribution of σ_r applied to the surface of cemented carbide ball with time is illustrated in Fig. 11. As is obvious from Fig. 11, σ_r is compression stress and has a maximum at the pole of cemented carbide ball. Furthermore it is seen that the stress in the neighborhood of pole becomes uniform and smooth as the heating period becomes longer.

3. Measurements of Pressure under High Temperature

The pressure sensor for measuring the generated pressure in the

Fig. 7 Temperature distribution of the cemented carbide ball and the alumina insulator

Fig. 8 Temperature distribution on the surface of cemented carbide ball in various heating periods

interface between the cemented carbide ball and the alumina insulator in section 2.4 must be

- (1) applicable to the measurements of high pressure under high temperature,
- (2) as thin as possible, and
- (3) as small as possible since the cemented carbide ball is small and the generated pressure differs according to the position.

As a result of investigating the pressure sensor to satisfy the above conditions in various respects, a sensor using the pressure dependence of $\alpha \rightleftharpoons \gamma$ transformation temperature of iron and $\alpha \rightleftharpoons \beta$ transformation temperature of cobalt was devised

Fig. 9 Stress distribution in a heating period of 15 s

Fig. 10 Stress distribution in a heating period of 50 s

Fig. 11 Distribution of σ_r on the surface of cemented carbide ball in various heating periods (spherical coordinates)

and made. Furthermore, the method for measuring the pressure under high-temperature by this sensor was experimentally investigated. The above transformation was detected by measuring the change of resistivity.⁽¹⁶⁾⁽¹⁷⁾ In this chapter, first the effect of heating rate on the transformation temperatures of iron and cobalt was experimentally investigated.

3.1 The effect of heating rate on the transformation temperatures of iron and cobalt

Considering the preceding condition for the pressure sensor, the transformation temperature of an iron sheet 8 mm long, 1 mm wide and 50 μm thick was measured from the change of resistivity at various heating rates between 20 $^{\circ}\text{C/s}$ ~ 98 $^{\circ}\text{C/s}$. The iron sheet was prepared electrolytically. In order to prevent the oxidation, the iron sheet was heated in a vacuum of 10^{-2} Pa. The relation between the heating rate and the transformation temperature is illustrated in Fig. 12. As is obvious from Fig. 12, in the above range of heating rates, the difference in transformation temperature depending on the heating rate is hardly recognized, and the transformation temperature is 906 $^{\circ}\text{C}$.

The cobalt sheet 8 mm long, 1 mm wide and 50 μm thick was prepared electrolytically, and its transformation temperature was measured in the above-mentioned range of heating rates. The relation between the transformation temperature and the heating time is illustrated in Fig. 12 in the same manner as iron. As is obvious from Fig. 12, the transformation temperature of cobalt doesn't depend on the heating rate, and it is 445 $^{\circ}\text{C}$.

It was found from the preceding experiments that the transformation temperatures of pure iron and pure cobalt could be considered to be

independent of the heating rate and to be constant.

3.2 Insulation resistance and thickness of MgO film

3.2.1 Measurements of the resistivity of MgO film

In order to use the preceding sheets of iron and cobalt as the pressure sensor, the insulation between the surface of cemented carbide ball and iron or cobalt is necessary to measure their resistivity. From the viewpoint of measuring pressure, the insulating material to be used for the above-mentioned purpose must have a sufficient effect of insulating even if it is thin. Furthermore, it is necessary for the insulating material and other materials not to diffuse into each other and interact under high temperature. It was found from the experimental investigations that MgO was a promising material for the insulating film which satisfied these requirements.

Then, the resistivity change of MgO film with temperature was investigated experimentally. MgO film of 2.5 μm thickness was formed on the mirror-finished disk of a cemented carbide by HF-sputtering. The specimen for measuring the resistivity of MgO film was made by coating a part of the surface of MgO film formed on a cemented carbide disk with a thin evaporated iron film. Heating the above specimen in a vacuum of 10^{-2} Pa, the resistivity of MgO film was measured in the range of 500 $^{\circ}\text{C}$ ~ 1000 $^{\circ}\text{C}$. The applied voltage between the cemented carbide disk and the iron film was 0.5 V. The results of measurements are illustrated in Fig. 13. The resistivity of MgO film is $10^{12} \Omega\cdot\text{m}$ at room temperature, decreases to $2.8 \times 10^6 \Omega\cdot\text{m}$ at 500 $^{\circ}\text{C}$, decreases to $2.0 \times 10^5 \Omega\cdot\text{m}$ at 850 $^{\circ}\text{C}$ as

Fig. 12 Relationship between heating rate and transformation temperature

Fig. 13 Relationship between resistivity of MgO film temperature

the temperature rises, above 850 °C the rate of decrease increases slightly, and is $5.0 \times 10^4 \Omega \cdot \text{m}$ at 1000 °C. As the alumina cylinder is made by hot-pressing, it is necessary to measure the effect of hot-pressing on the resistivity of MgO film. The results of measurements are illustrated in Fig.13. As is obvious from Fig.13, the resistivity of MgO film decreases one order by hot-pressing.

3.2.2 Thickness of an insulating film required to measure the electric resistance of iron sheet and cobalt sheet.

For the preceding purpose of experiments, the thinner the insulating film, the better. But it is necessary to determine the thickness of insulating film considering the thickness of the pressure sensor and the distance for measuring a voltage drop.

Then the layer-built structure

$$V_{BC} = \frac{l_0 I}{2\pi\sigma_1 l_1 (l_0 + l_1)} + \frac{I}{\pi\sigma_1} \int_0^\infty \frac{\left[\frac{(\sigma_2 - \sigma_3) \exp\{-2\lambda(\delta_1 + \delta_2)\}}{(\sigma_2 + \sigma_3) + (\sigma_1 - \sigma_2)} \times \exp(-2\lambda\delta_1) / ((\sigma_1 + \sigma_2)[J_0(\lambda l_1) - J_0(\lambda(l_0 + l_1))]) d\lambda \right]}{\left[1 - \frac{(\sigma_2 - \sigma_3) \exp\{-2\lambda(\delta_1 + \delta_2)\}}{(\sigma_2 + \sigma_3) + (\sigma_1 - \sigma_2)(\sigma_2 - \sigma_3)} \times \exp(-2\lambda\delta_2) / \{(\sigma_1 + \sigma_2)(\sigma_2 + \sigma_3) - (\sigma_1 - \sigma_2) \exp(-2\lambda\delta_1) / ((\sigma_1 + \sigma_2))\} \right]} d\lambda \quad (6)$$

When the insulating film is a perfect insulator, the voltage difference V'_{BC} between points B and C is given by equation (7) substituting $\sigma_2 = 0$ into equation (6).

$$V'_{BC} = \frac{l_0 I}{2\pi\sigma_1 l_1 (l_0 + l_1)} + \frac{I}{\pi\sigma_1} \int_0^\infty \frac{\exp(-2\lambda\delta_1)}{1 - \exp(-2\lambda\delta_1)} [J_0(\lambda l_1) - J_0(\lambda(l_0 + l_1))] d\lambda \quad (7)$$

When taking the thickness δ_1 of Fe pressure sensor and the distance for measuring a voltage drop l_0 and l_1 to be 0.01 mm, 1 mm and 0.1 mm respectively, the ratio V_{BC}/V'_{BC} was calculated changing the thickness δ_2 of MgO film. The results are illustrated in Fig.15. It is obvious from Fig.15 that V_{BC}/V'_{BC} is equal to 0.99999 at 1000 °C. In other words, it is sufficient to take the thickness of MgO film to be 0.2 μm in order to measure the value of V_{BC} with an accuracy of 0.001 %.

Fig. 14 Calculation model for measuring the resistivity of an iron sheet on the thin layer of MgO formed on the surface of a cemented carbide ball

(see Fig.14) is considered, in which an insulating film of conductivity σ_2 with thickness δ_2 is placed on the surface of a semi-infinite solid of conductivity σ_3 and the pressure sensor of conductivity σ_1 with thickness δ_1 is placed on the insulating film. In Fig.14, the voltage difference V_{BC} between points B and C in a straight line connecting point A to D produced by passing current I from point A to D can be obtained as follows. A structure symmetric with respect to the surface of pressure sensor is imagined. It is assumed that the dielectric constants of a pressure sensor, an insulating film and a semi-infinite solid are ϵ_1 , ϵ_2 , ϵ_3 respectively, and the charges Q and $-Q$ are at points A and D respectively. Then, solving the electrostatic field problem of obtaining the potential difference between points B and C, the voltage difference V_{BC} is obtained. It is given by equation (6).

Fig. 15 Relationship between V_{BC}/V'_{BC} and thickness of MgO film under high temperature

3.3 Experimental method and measured results

3.3.1 Experimental method

The arrangements of a pressure sensor, MgO film and a thermocouple for the measurement of the generated pressure in the interface between the cemented carbide ball and alumina are illustrated in Fig. 16. The diameters of cemented carbide ball and alumina cylinder were 3.2 mm and 10 mm respectively, which are equal to those of an example of the calculation. The shape and dimensions of the sheet of pure iron and pure cobalt were equal, i.e., 5 mm long, 0.5 mm wide and 0.01 mm thick. Though the minimum thickness of MgO film required to measure the resistivity of pressure sensor with an accuracy of 0.001 % was 0.2 μm from the calculated results in section 3.2.2, the thickness of MgO film was taken to be 2.5 μm considering various cases of the decrease in the resistivity of the insulating film described above. Pt/Pt-10 % Rh-thermocouple was used as a thermometer, because the effects of pressure on the emf of this thermocouple were measured by Hanneman⁽¹⁹⁾. The alternating voltage induced by the field in the thermocouple and the lead wire for measuring the resistance was removed using a low-pass filter. The procedure of making the high-pressure generating cylinder for the measurement of pressure illustrated in Fig. 16 is as follows.

(1) The alumina cylinder with a diameter 10 mm and a height of 8 mm was sintered by hot-pressing at 1400 °C, 30 MPa for 6 min, where the center of cemented carbide ball was put in the axis and at distance 2 mm from the end of cylinder.

(2) The end of the alumina cylinder on the side of the cemented carbide ball was lapped using the diamond abrasive grain with an average diameter of 50 μm until a diameter of the circular section of the cemented carbide ball becomes 2 mm.

(3) The above section was mirror-finished using an impalpable powder of diamond with an average diameter of 1 μm .

(4) The above mirror-finished circular section of the cemented carbide ball was coated with MgO film of 3 mm diameter and 2.5 μm thickness. Furthermore, the thickness of MgO film was measured with a high-precision film gap measuring instrument.

(5) A pressure sensor was placed on the above-mentioned MgO film coinciding the center of pressure sensor with that of the preceding circular section and putting the distance $l_0 = 1$ mm for measuring the voltage drop in the circle with a diameter of 1.2 mm.

(6) 0.16 g of alumina powder was packed in uniform thickness of a green compact on the side where the pressure sensor was placed, the alumina cylinder with a diameter 10 mm and a height of 4 mm was put on it, and they were sintered in one united body by hot-pressing at 1250 °C, 30 MPa for 3 min in a vacuum.

Both ends of the above pressure-generating cylinder for measuring the pressure were restrained in the direction of the axis by pressing at 50 MPa through the alumina anvil which equals in diameter to the above cylinder, so that the boundary condition might be in agreement with the condition assumed in the calculation. The frequency of current flowing in an induction coil was 170 kHz. Furthermore, the current flowing through a pressure sensor for the measurement of the electrical resistance was 25 mA.

3.3.2 Measured results

The time-change in temperature and resistivity of the iron and cobalt pressure sensor after the start of heating the pressure-generating cylinder by induction is illustrated in figures 17 and 18 respectively. It is seen from Fig. 17 that the resistivity of iron increases as the temperature rises, the rate of increase increases at 735 °C and decreases slightly above 740 °C. As is already clarified, since the resistivity of iron in γ -phase is larger than in α -phase,⁽¹⁶⁾⁽¹⁷⁾ the preceding clear change of the resistivity shows the generation of transformation from α -phase to γ -phase. Then the $\alpha \rightarrow \gamma$ transformation

Fig. 16 Arrangement of a pressure sensor and circuit for the measurements of electrical resistance and temperature of iron sheet and cobalt sheet

temperature was taken to be a temperature at the start of the change of resistivity. Thereby the $\alpha \rightarrow \gamma$ transformation temperature of 735°C is the value at 2.6 GPa from the pressure-temperature phase diagram⁽¹⁵⁾ of iron. It is seen from Fig. 18 that the resistivity of cobalt increases as the temperature rises, in the region of 478°C - 488°C the rate of increase remarkably decreases, and above 488°C it increases again as the temperature rises. Thereby it is found that $\alpha \rightarrow \beta$ transformation starts at 478°C and finishes at 488°C. The transformation temperature was taken to be a temperature at the start of transformation. The transformation temperature 478°C is the value at 0.9 GPa from the pressure-temperature phase diagram⁽¹⁵⁾ of cobalt.

As is evident from the above, when induction heating the cemented carbide ball located at the center of the alumina cylinder to generate a pressure by restraining thermal expansion, the pressure is generated in the interface between the cemented carbide ball and alumina, and the pressures of 0.9 GPa and 2.6 GPa are generated at 478°C and 735°C respectively.

The calculated values of σ_R applied to the surface of cemented carbide ball are 1.1 GPa and 1.9 GPa at the preceding temperatures 478°C and 735°C respectively, which is closer to the measured value. Thereby applied to the interface between the conductor ball and the insulator is obtained using the

method of calculation described in section 3.2, which almost agrees with the pressure generated in the above interface.

The limit of pressure generated by the high-temperature and high-pressure generating apparatus in general use is 10 GPa⁽¹⁾⁽⁵⁾⁽⁷⁾ in the apparatus of piston-cylinder type, belt type and multi-anvil type, and 20 GPa⁽⁶⁾ in the apparatus of high-compression belt type improved by Bundy. The apparatus for generating high pressure by restraining thermal expansion has the advantage of obtaining high pressure and high temperature simultaneously without the apparatus of a large size. But it is a matter of course that there is a limit in the generated pressure of the devised apparatus, because the mechanical strength and the modulus of longitudinal elasticity of the insulator decreases owing to the temperature rise and there is a limit in the coefficient of thermal expansion of conductor. Trying to calculate the generated pressure on the combination of materials considered now, the high pressure of 6 GPa is obtained at 1500°C in the case of using alumina and iron as insulator and conductor respectively. It is considered from the calculation that the limit of pressure generated by the apparatus devised in this paper is not above that of the conventional apparatus when a conductor ball is in a state of solid.

4. Conclusions

A method for generating a high pressure by restraining thermal expansion and expansion on melting was proposed. The methods of calcu-

Fig. 17 Temperature and electrical resistance changes of the pressure sensor of iron depending on heating time

Fig. 18 Temperature and electrical resistance changes of the pressure sensor of cobalt depending on heating time

lating the pressure generated by the above method and measuring the pressure under high-temperature were proposed. After the possibility of the method of generating a high pressure proposed in this paper was investigated by the numerical calculations and experiments on one example, the following results became evident.

(1) When a cemented carbide ball with a diameter of 3.2 mm was heated by induction which was centered in the alumina cylinder with a diameter 10 mm and a height of 12 mm, the calculated value of σ_r generated at the pole of cemented carbide ball was 1.1 GPa at 480°C and 1.9 GPa at 735°C.

(2) The pressure generated in the high-pressure apparatus which had the same size as the example of calculation was measured using the pressure dependence of the transformation temperatures of iron and cobalt, which was 0.9 GPa at 478°C and 2.6 GPa at 735°C.

(3) The generated pressures estimated by the method of calculation proposed in this paper agree with the measured values.

(4) The method of the pressure measurement using the pressure dependence of the transformation temperature of iron and cobalt was applicable to high-pressure measurements under high-temperature.

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