Elastic Anisotropy and its Temperature Variation of Single Crystals of the High Elasticity Alloy "Dia-flex" in the Co-Fe-Ni-Cr-W-Mo System*

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Temperature dependences of thermal expansion, Young's modulus and rigidity modulus, and mechanical strength at room temperature were measured with single crystals and annealed polycrystals of high elasticity alloy "Dia-flex" with the *fcc* structure: The alloy "Dia-flex", composed mainly of 38.0% Co, 22.4% Fe, 16.5% Ni, 12.0% Cr, 4.0% W and 4.0% Mo showed the Young's modulus of 23×10^5 kg/cm², the elastic limit of 120 kg/mm² and tensile strength of 250 kg/mm² in the rolling direction. From the data obtained, the volume compressibility, principal elastic parameters, principal elastic constants, and Young's modulus and rigidity modulus in the three principal orientations of the single crystals were calculated. The results thus obtained are as follows: the Young's moduli at 20°C are 11.68×10^5 kg/cm², 22.49×10^5 kg/cm² and 33.78×10^5 kg/cm², and the rigidity moduli at 20°C, 12.01×10^5 kg/cm², 6.02×10^5 kg/cm² and 5.13×10^5 kg/cm² in the $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ directions of the single crystals, showing a high anisotropy of the elastic moduli. The anisotropy of the temperature coefficients of elastic moduli is also very high.

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I. Introduction

The watch main spring requires high tensile strength, elastic limit, Young's modulus and corrosion resistivity. As its materials, alloys of multicomponent face-centered cubic structure (for example, Elgiloy⁽¹⁾) which consist principally of Co, Fe, Ni, Cr, W and Mo are most extensively used after appropriate working and heat treatments. Since these alloys have a large elastic crystal anisotropy, a great deal of investigations have hitherto been concerned with the methods of working and heat treatments so as to make the predominant direction of elasticity meet with a direction required in practical applications as much as possible⁽²⁾. However, it seems that there is no literature available in which the elastic modulus and anisotropy have been determined with the single crystals.

The present authors produced single crystals of the high-elasticity alloy "Dia-flex" in the Co-Fe-Ni-Cr-W-Mo system⁽³⁾, the elastic data in the predominant direction were 23×10^5 kg/cm² in Young's modulus, 120 kg/mm² in elastic limit and 250 kg/mm² in tensile strength. Measurements were also made of the

Young's modulus and its temperature variation for the single crystals in the annealed state. On the basis of these results, the Young's modulus for the polycrystal was obtained using various theoretical relations and was compared with the measured values. Also, the rigidity modulus and mechanical properties at room temperature were measured.

II. Specimens and Method of Measurement

1. Specimens

For the preparation of single crystals, a 2 mm wire made of a high-elasticity alloy (38.0% Co, 22.4% Fe, 16.5% Ni, 12.0% Cr, 4.0% Mo, 4.0% W, 1.2% Mn, 1.0% Ti and 0.8% Si) was melted in vacuum with an alumina crucible 3 mm in inner diameter and then processed by Tamman-Bridgman's method⁽⁴⁾. After a number of trial preparations, it was found that this alloy, as in the case of other *fcc* alloys, shows the most easily obtainable orientation to be the $\langle 100 \rangle$ direction⁽⁵⁾ and the optimal descending rate for the growth of the single crystals, 5.0 mm/min. However, it was very difficult to produce single crystals with the $\langle 110 \rangle$ and $\langle 111 \rangle$ orientations by this method, so that the crystals were connected and grown at an appropriate angle with the crystal as the seed. In this case, the optimal descending rate was 4.0 mm/min and 3.7 mm/min, respectively. Since in some cases the single crystals so prepared contained bubbles, those which deviated from their proper values were excluded from measurements on the basis of the measured values of density. In this way, cylindrical single crystals 15~20 cm long and about 3 mm in diameter were prepared, and the crystal specimens were all standardized to the length of 12 cm for the convenience of measure-

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(1) Manufactured by the Elgin National Watch Co. U.S.A. 40Co-25Cr-22Ni-0.03Be-(0.15~0.20)C-2Mn-bal Fe system alloys.

(2) M. F. Straumann: Soc. Suisse de Chronom., (1954), 209; M. E. Dubois: Soc. Suisse de Chronom., (1954), 215; J. Satô, K. Ikeda and N. Kamata: J. Japan Chronom. Soc., **12** (1961), 39, etc.

(3) H. Masumoto, H. Saitô and T. Kobayashi: Japan patent No. 230713 (Dec. 14, 1956)

(4) M. Yamamoto: Sci. Rep. Tôhoku Imp. Univ., **29** (1940), 113; J. Japan Inst. Metals, **4** (1940), 368.

(5) M. Yamamoto: J. Japan Inst. Metals, **4** (1940), 27, 168, 222.

ment. The orientation was determined by the light-figure method⁽⁴⁾⁽⁶⁾ after corroding the specimens in a boiling supersaturated ferric chloride solution for 10 min or in dilute aqua regia for 2 min. The elastic modulus of a polycrystal specimen about 3 mm in diameter and the rigidity modulus of a polycrystal specimen about 0.5 mm in diameter were measured. In both cases, the alloy melts were solidified to obtain quasi-isotropic specimens, with care to suppress the growth of the crystals. Subsequently, to eliminate strains, the specimens were annealed at the temperature of 800°C at which recrystallization did not take place in vacuum. Fig. 1 shows a stereographic projection of orientation distributions of all the single crystal specimens⁽⁷⁾, in which only the specimens numbered in the figure were measured and their orientations are illustrated in Table 1.

Fig. 1 Stereographic projection of orientation of single crystals of Co-Fe-Ni-Cr-W-Mo alloy. (Specimen Nos. 1~10)

Table 1 Direction cosines of the rod axis of single crystals of Co-Fe-Ni-Cr-W-Mo alloy

Specimen No.	a_1	a_2	a_3
1	54.21	55.30	48.28
2	54.00	56.30	47.52
3	55.30	55.30	53.14
4	45.30	84.30	45.02
5	46.25	85.00	44.02
6	82.30	83.50	9.58
7	88.30	89.00	1.48
8	87.00	88.30	2.48
9	89.00	89.50	1.06
10	89.00	89.00	1.31

2. Method of measurement

The mean values of linear thermal expansion coefficients were first determined over the temperature range 0°~800°C using the Super-Invar type vertical dilatometer⁽⁸⁾ and the ordinal horizontal

dilatometer, and the density was measured by weighing in water. The Young's moduli were calculated from the natural frequencies (600~800 c/s) of the specimen excited by the vibrator-controlled oscillator system⁽⁹⁾ at -50°~300°C. For this calculation the measured values of thermal expansion coefficient and density were also used. The rigidity modulus was measured at 10°~50°C on a polycrystal specimen of 0.5 mm diameter using the torsional pendulum method⁽¹⁰⁾. The tensile strength was measured from the annealed specimens with an Instron's testing machine under a load of 200~2000 kg.

III. Experimental Results and Considerations

1. Thermal expansion

Three specimens having the nearest orientations to $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$, respectively, were used to measure the thermal expansions at temperatures between 0° and 800°C, and the results of measurements are shown in Fig. 2. As can be seen from the figure, the thermal expansion is isotropic and the slope of the curve changes clearly at the temperature of 558°C; this is probably because a slight amount of the remaining magnetism disappears at this temperature. The curves on both sides of these knees are almost linear, and the thermal expansion coefficients are 13.40×10^{-6} and 15.71×10^{-6} in the low and high temperature ranges, respectively. These values are in good agreement with those for the polycrystal specimen.

Fig. 2 Thermal expansion $\Delta l/l$ of single crystals of Co-Fe-Ni-Cr-W-Mo alloy

- (6) M. Yamamoto : J. Japan Inst. Metals, 5 (1941), 214; J. Appl. Phys., Japan, 10 (1940), 199.
- (7) M. Yamamoto : Met. Phys., 6 (1960), 118; C. S. Barrett : *Structure of Metals*, (1952), p. 26, The stereographic projection, Birmingham, England.
- (8) H. Masumoto and T. Kobayashi : Sci. Rep. RITU, A-2 (1950), 856; J. Japan Inst. Metals, 12 (1948), No. 6-1.

- (9) Y. Shirakawa and I. Oguma : J. Japan Inst. Metals, 24 (1960), 63.
- (10) H. Masumoto and H. Saitô : J. Japan Inst. Metals, 16 (1952), 126; P. Chevenard : Trav. et Mém du Bur. Int. des Poids et Mesures, 17 (1927), 44.

2. Young's modulus and rigidity modulus

Fig. 3 indicates temperature changes of Young's modulus E , showing a large anisotropy of E . However, since its value at an arbitrary direction cannot be obtained only from the measured values of E , the following relation for the isometric system crystals⁽¹¹⁾ is applied:

$$\frac{1}{E} = S_{11} - 2 \left(S_{11} - S_{12} - \frac{1}{2} S_{44} \right) \times (\alpha_1^2 \alpha_2^2 + \alpha_2^2 \alpha_3^2 + \alpha_3^2 \alpha_1^2), \quad (1)$$

Fig. 3 Temperature variations of Young's modulus E of single crystals and a polycrystal of Co-Fe-Ni-Cr-W-Mo alloy

where S_{ij} is the elastic parameter and α_i, α_j are the direction cosines of the rod axis with regard to the crystal axes. And $\sum \alpha_i^2 \alpha_j^2$ is the orientation function, its value being 0, 1/4 and 1/3 in the $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ directions, respectively. Fig. 4 represents the relationship between $1/E$ and $3 \sum \alpha_i^2 \alpha_j^2$, in which the measured points are nearly on a straight line. Accordingly, assuming eq. (1) to be valid, the values of S_{11} and $S_{11} - S_{12} - (1/2) S_{44}$ can be calculated from the measured values of E using a least square method. Moreover, the E can be calculated in the $\langle 110 \rangle$ plane which contains three principal axes as follows:

$$(\alpha_1^2 \alpha_2^2 + \alpha_2^2 \alpha_3^2 + \alpha_3^2 \alpha_1^2) = \frac{1}{4} (1 + 2 \cos^2 \theta - 3 \cos^4 \theta), \quad (2)$$

(11) W. Voigt : *Lehrbuch der Kristallphysik* (B.G. Teubner Leipzig), (1928), 739, 741, 410; W.A. Wooster: *Crystal Physics* (Cambridge), (1938), 1.

Fig. 4 Relations between reciprocals of Young's modulus E and rigidity modulus G , and the orientation function $3(\alpha_1^2 \alpha_2^2 + \alpha_2^2 \alpha_3^2 + \alpha_3^2 \alpha_1^2)$ of single crystals of Co-Fe-Ni-Cr-W-Mo alloy

Fig. 5 Temperature variations of calculated values of elastic parameters S_{11} and $S_{11} - S_{12} - (1/2) S_{44}$ of single crystals of Co-Fe-Ni-Cr-W-Mo alloy

where θ is an angle between the $\langle 001 \rangle$ direction and the rod axis of the specimen. By substituting this equation into eq. (1), the value of E at an arbitrary direction can be calculated. Figs. 5, 6 and 7 show the temperature variation of the calculated values of S_{11} and $S_{11} - S_{12} - (1/2) S_{44}$, the temperature variation of the calculated values of E in the three principal directions and the relationship between E and orientations in the $\langle 110 \rangle$ plane at -50° , 20° and 300°C , respectively. As shown in Fig. 6, E decreases almost linearly in a given direction in the temperature range $-50^\circ \sim$ about 20°C and then parabolically; at each temperature the value is greatest in the $\langle 111 \rangle$ direction and smallest in the $\langle 100 \rangle$ direction. This result agrees well with the relation for the Young's modulus in the principal orientations as follows:

$$E_{\langle 111 \rangle} > E_{\langle 110 \rangle} > E_{\langle 100 \rangle}. \quad (3)^{(2)}$$

Especially, E in the $\langle 111 \rangle$ direction is $33.78 \times 10^5 \text{ kg/cm}^2$ (20°C) and the above relation is noteworthy in view of the fact that the watch main springs usually have

Fig. 6 Temperature variations of calculated values of Young's modulus E in three principal orientations of single crystals and a polycrystal of Co-Fe-Ni-Cr-W-Mo alloy, and elastic anisotropies $E_a(E_{\langle 111 \rangle}/E_{\langle 100 \rangle})$ and $G_a(G_{\langle 100 \rangle}/G_{\langle 111 \rangle})$

Fig. 7 Calculated values of Young's modulus E , rigidity modulus G and their temperature coefficients e and g of various orientations in (110) plane of single crystals of Co-Fe-Ni-Cr-W-Mo alloy

a $\langle 112 \rangle$ (110) orientation in the rolling direction and an E -value of $23 \times 10^5 \text{ kg/cm}^2$ (2). In Fig. 6 are also shown the temperature vs. $E_{\langle 111 \rangle}/E_{\langle 100 \rangle}$ curves; its values are fairly large compared with ordinary pure metals of *fcc* structure⁽¹²⁾, 2.89 at 20°C and 3.18 at 300°C . The mean temperature coefficients $e_{0^\circ \sim 40^\circ\text{C}}$ of E in all the orientations in the (110) plane at $0^\circ \sim 40^\circ\text{C}$ are shown in Fig. 7, and those three principal orientations at $0^\circ \sim 40^\circ\text{C}$ and $-50^\circ \sim 300^\circ\text{C}$ are given in Table 2.

(12) on Fe and Ni for example, M. Yamamoto : J. Japan Inst. Metals, 6 (1942), 331,

The values of E for the quasi-polycrystal can be derived from ReuB' relation⁽¹³⁾

$$E_p = \frac{5}{3S_{11} + 2S_{12} + S_{44}} \quad (4)$$

by applying the mean value of S_{ij} for strain components under the assumption that the stress is equal for all crystallites. A comparison between the values of E_p calculated by the above relation and the measured values of E_p for the quasi-polycrystal specimen slow-cooled after melting is shown in Fig. 6, both being in good agreement.

As shown in Fig. 8, the measured values of rigidity modulus G decreases almost linearly with temperature, as is the case with E . G at room temperature and its temperature coefficient g are presented in Table 2.

Fig. 8 Temperature change of measured and calculated values of rigidity modulus G and calculated values of volume compressibility κ of Co-Fe-Ni-Cr-W-Mo alloy

Next, the volume compressibility κ must be known to determine all the values of S_{ij} . From the relation for κ , E and G

$$K = \frac{3(3G - E)}{EG}, \quad (5)$$

the values of κ were calculated and are shown in Table 2. Since there is a relation

$$K = 3(S_{11} - 2S_{12}), \quad (6)^{(11)}$$

between κ and S_{ij} , S_{12} was obtained using the values of S_{11} , shown in Fig. 5, and then S_{44} was calculated from the value of $S_{11} - S_{12} - (1/2)S_{44}$. The result thus obtained is summarized in Table 3. The principal elastic constant C_{ij} can be derived from the following relations⁽¹¹⁾:

$$\left. \begin{aligned} C_{11} &= \frac{S_{11} + S_{12}}{(S_{11} - S_{12})(S_{11} + 2S_{12})} \\ C_{12} &= \frac{-S_{12}}{(S_{11} - S_{12})(S_{11} + 2S_{12})} \\ C_{44} &= \frac{1}{S_{44}} \end{aligned} \right\} \quad (7)$$

The values of C_{ij} at 20°C , calculated from the above relations, are tabulated in Table 3, from which it can be predicted that Cauchy's relation $C_{12} = C_{44}$ ⁽¹⁴⁾ does

(13) A. ReuB : Z. angew. Math. U. Mechn., 9 (1929), 49.

(14) W. Voigt : loc. cit., 410.

Table 2 Calculated values of Young's modulus E , rigidity modulus G and their temperature coefficients e and g in three principal orientations of single crystals and volume compressibility κ of Co-Fe-Ni-Cr-W-Mo alloy

	E	G	κ	e		g
	($\times 10^5$ kg/cm ²)		($\times 10^{-5}$ cm ² /kg)	($\times 10^{-5}$)		
	20°C			0°~40°C	-50°~300°C	0°~40°C
$\langle 100 \rangle$	11.68	12.01	—	-36.20	-39.32	-12.73
$\langle 110 \rangle$	22.49	6.02	—	-23.25	-29.26	-29.97
$\langle 111 \rangle$	33.78	5.13	—	-14.76	-20.16	-33.00
polycrystal	18.99	6.71	0.0270	-18.22	-30.48	-28.40

Table 3 Calculated values of elastic parameters S_{ij} and elastic constants C_{ij} at 20°C of Co-Fe-Ni-Cr-W-Mo alloy

S_{ij}	($\times 10^{-5}$ cm ² /kg) 20°C	C_{ij}	($\times 10^5$ kg/cm ²) 20°C
S_{11}	0.0856	C_{11}	43.00
S_{12}	-0.0383	C_{12}	34.82
S_{44}	0.0832	C_{44}	12.01

not hold in this case⁽¹⁵⁾. Since G and S_{ij} for the principal directions in the isometric system crystal specimen have a relation

$$\frac{1}{G} = S_{44} + 4 \left(S_{11} - S_{12} - \frac{1}{2} S_{44} \right) \times (a_1^2 a_2^2 + a_2^2 a_3^2 + a_3^2 a_1^2), \quad (8)$$

the values of G for various orientations and temperatures can be calculated by substituting the previously determined values of S_{ij} into the above relation. The results thus obtained are shown in Fig. 7 and Table 2, from which it is clear that G is greatest in the $\langle 100 \rangle$ direction and smallest in the $\langle 111 \rangle$ direction. Yamamoto⁽¹²⁾ reported a similar finding on Fe and Ni of the cubic crystal system. The relationship between $1/G$ and the direction function at 20°C is shown in Fig. 4, and the curves are almost linear. Also, the mean temperature coefficient, $g_0 \sim 40^\circ\text{C}$, of G for the temperatures of $0^\circ \sim 40^\circ\text{C}$ is greatest in the $\langle 100 \rangle$ direction and smallest in the $\langle 111 \rangle$ direction, as is the case with E . Although the anisotropy of G , $G_{\langle 100 \rangle}/G_{\langle 111 \rangle}$, could not be measured over an extensive temperature range, the value showed a gradual increase such as 2.34 at 20°C and 2.36 at 50°C, as shown in Fig. 6, in a way similar to the case of E . The following relation⁽¹³⁾ holds between G_p and S_{ij} :

$$G_p = \frac{5}{4S_{11} - 4S_{12} + 3S_{44}}. \quad (9)$$

The value of G_p calculated by this relation are shown in Fig. 8, and the values do not differ greatly from the measured ones in the quasi-isotropic specimen.

Table 4 Mechanical properties of single crystals and a polycrystal of the Co-Fe-Ni-Cr-W-Mo alloy

Specimen No.	Elastic limit (permanent strain, 0.003%) (kg/mm ²)	Yield point (kg/mm ²)	Tensile strength (kg/mm ²)
9	21.40	24.65	40.80
4	18.60	18.93	34.00
2	22.01	26.27	45.27
polycrystal	19.10	19.20	35.70

3. Mechanical strength

In Table 4 are shown the elastic limit, yield point and tensile strength at 20°C in the annealed state for No. 9 alloy specimen having an orientation near $\langle 100 \rangle$, No. 4 specimen near $\langle 110 \rangle$, No. 2 specimen near $\langle 111 \rangle$ and a polycrystal specimen. As can be seen from the table, their values near the $\langle 111 \rangle$ direction are higher than those near the other directions.

IV. Conclusion

The thermal expansion at $0^\circ \sim 800^\circ\text{C}$, the Young's modulus at $-50^\circ \sim 300^\circ\text{C}$, the rigidity modulus at $10^\circ \sim 50^\circ\text{C}$ and mechanical properties at 20°C were measured with cylindrical single crystals of a high-elasticity alloy "Dia-flex" in the Co-Fe-Ni-Cr-W-Mo system which was slowly cooled from 800°C. The results obtained are as follows:

(1) The optimum descending rate for growth of the single crystals is 5.0, 4.0 and 3.7 mm/min in the $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ directions, respectively.

(2) The thermal expansion curves obtained for the single crystals at $0^\circ \sim 800^\circ\text{C}$ are all isotropic and show a distinct knee in the vicinity of 558°C. The mean thermal expansion coefficients in the temperature range above and below the knee are 15.71×10^{-6} and 13.40×10^{-6} , respectively.

(3) The calculated values of Young's modulus in the three principal directions and the polycrystal specimen at 20°C are $E_{\langle 100 \rangle} = 11.68 \times 10^5$ kg/cm², $E_{\langle 110 \rangle} = 22.49 \times 10^5$ kg/cm², $E_{\langle 111 \rangle} = 33.78 \times 10^5$ kg/cm² and $E_p = 18.99 \times 10^5$ kg/cm², among which the value of E_p is almost consistent with the measured one. $E_{\langle 111 \rangle}/E_{\langle 100 \rangle}$ is 2.89 at 20°C and 3.18 at 300°C, showing the increases with temperature.

(4) The temperature variation of Young's modulus resembles each other in each orientation, and the mean temperature coefficient at $0^\circ \sim 40^\circ\text{C}$ in the three

(15) on Al, G. N. Kamm and G. A. Alers : J. Appl. Phys., 35 (1964), 327; on Fe-Ni, G. A. Alers and J. R. Neighbours : J. Phys. Chem. Solids, 13 (1960), 40; on Ta, W, Mo, F. M. Featherston and J. R. Neighbours : Phys. Rev., 130 (1963), 1324; other survey in R. F. S. Hearmon : Rev. Mod. Phys., 18 (1946), 432.

principal directions and the polycrystal specimen is $e_{\langle 100 \rangle} = -36.20 \times 10^{-5}$, $e_{\langle 110 \rangle} = -23.25 \times 10^{-5}$, $e_{\langle 111 \rangle} = -14.76 \times 10^{-5}$ and $e_p = -18.22 \times 10^{-5}$, respectively.

(5) The principal elastic parameter at 20°C is $S_{11} = 0.0856 \times 10^{-5} \text{ cm}^2/\text{kg}$, $S_{12} = -0.0383 \times 10^{-5} \text{ cm}^2/\text{kg}$ and $S_{44} = 0.0832 \times 10^{-5} \text{ cm}^2/\text{kg}$.

(6) The principal elastic constant at 20°C is $C_{11} = 43.00 \times 10^5 \text{ kg/cm}^2$, $C_{12} = 34.82 \times 10^5 \text{ kg/cm}^2$ and $C_{44} = 12.01 \times 10^5 \text{ kg/cm}^2$. Cauchy's relation does not hold for this alloy.

(7) The rigidity modulus at 20°C in the three principal directions and the polycrystal specimen was calculated to be $G_{\langle 100 \rangle} = 12.01 \times 10^5 \text{ kg/cm}^2$, $G_{\langle 110 \rangle} = 6.02 \times 10^5 \text{ kg/cm}^2$, $G_{\langle 111 \rangle} = 5.13 \times 10^5 \text{ kg/cm}^2$ and $G_p = 6.71 \times 10^5 \text{ kg/cm}^2$, respectively, among which the values of G_p is almost consistent with the measured one. $G_{\langle 100 \rangle}/G_{\langle 111 \rangle}$ is 2.34 at 20°C and 2.36 at 50°C, showing a gradual increase with temperature.

(8) The mean temperature coefficient of rigidity modulus at 0°~40°C in the three principal directions and the polycrystal specimen is $g_{\langle 100 \rangle} = -12.73 \times 10^{-5}$,

$g_{\langle 110 \rangle} = -29.97 \times 10^{-5}$, $g_{\langle 111 \rangle} = -33.00 \times 10^{-5}$ and $g_p = -28.40 \times 10^{-5}$, respectively.

(9) The volume compressibility at 20°C is calculated to be $0.0270 \times 10^{-5} \text{ cm}^2/\text{kg}$.

(10) The tensile strengths at 20°C in the directions near $\langle 100 \rangle$, $\langle 110 \rangle$ and $\langle 111 \rangle$ and the polycrystal specimen are 40.80, 34.00, 45.27 and 35.70 kg/mm², respectively.

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