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Study on the applicability of the Friedel relation with dislocation velocity-stress exponent

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It was investigated at 77–178 K whether the Friedel relation is appropriate for the interaction between a dislocation and the impurity in KCl:Mg^{2+} (0.035 mol% in the melt) single crystal. This was carried out by fitting of $m_{\varepsilon=0}$ to the temperature versus dislocation velocity-effective stress exponent, m^*, curves with respect to the two models: one is the Fleischer's model taking account of the Friedel relation and the other the Fleischer's model. The former model is termed the F-F. $m_{\varepsilon=0}$ is the m value at zero strain. m is a dislocation velocity-applied stress exponent. The m^* for the F-F was examined from

$$m^* = 2F_0 b\{(T_c/T)^{1/2} - 1\}/(3kT_c)$$

where F_0 is the force acted on the dislocation at 0 K, b is the magnitude of the Burgers vector, k is the Boltzmann's constant and T_c is the critical temperature T, at which the effective stress due to the impurities is zero. As a result, it was found that the $m_{\varepsilon=0}$ values tend to increase with decreasing temperature and further approach to the m^* for the Fleischer's model in contrast to the F-F. That is, the interaction between a dislocation and the impurity in the specimen could be approximated to the Fleischer's model rather than to the F-F. Similar result was also deduced on the basis of the data analyzed in terms of strain-rate sensitivity versus stress decrement due to oscillation. Consequently, it was considered that the Friedel relation is inappropriate for the specimen within the temperature range. *© 2003 Kluwer Academic Publishers*

1. Introduction

When alkali halide crystals are doped with divalent impurities, the impurities are bound to positive ion vacancies in the form of a dipole. The divalent impurity-vacancy dipole is abbreviated to I-V dipole. Then asymmetrical distortions are produced around the I-V dipoles and interact strongly with mobile dislocations [1]. The solution hardening is named "rapid hardening," which can be distinguished from "gradual hardening" due to the defects of cubic symmetry (e.g., substitutional atoms in cubic crystals, monovalent substitutional impurities in NaCl, interstitials in face-centered cubic metals, and F-centers (additive coloring) in KCl) [2, 3]. In view of the different types of atomic defects, solution hardening may be divided into the two classes.

It is well known that the Friedel relation [4] between the effective stress and the average length of dislocation segments is exact for most weak obstacles to dislocation motion at low concentration of the obstacles. By using a digital computer, this was clarified by Foreman and Makin [5]. It is investigated in this paper whether the Friedel relation is exact for readily available KCl:Mg^{2+} crystals concerned with the above-mentioned rapid hardening. A large number of investigations on strength of materials have been made with alkali halide crystals so far [6–11]. Alkali halide contained additive (i.e., KCl:Mg^{2+}), therefore, is an excellent material for an investigation of mechanical properties.

This study is carried out by the use of dislocation velocity-effective stress exponent expressed by [12]

$$\upsilon = A\tau^{m^*} \tag{1}$$

where υ is the average velocity of dislocation, A and m^* are constants for a given material and temperature, and τ is the effective shear stress due to the impurities. Equation 1 is regarded as an empirical relationship [13, 14]; nevertheless, m^* is useful for investigating the behavior of thermally activated dislocation motion in various materials (e.g., NaCl contained Mg^{2+}, Ca^{2+}, Sr^{2+}, and Ba^{2+} [15], body-centered cubic metals [16], Nb [17], LiF:Mg^{2+} [18], and binary iron-base alloys contained Co, Cr, Al, Si, Ni and Mn, respectively [19]). From m^*, rate-controlling mechanism has been examined [20–22].

2. Experimental procedure

KCl:Mg^{2+} (0.035 mol% in the melt) single crystals were deformed by compression along ⟨100⟩ axis and the ultrasonic oscillatory stress was applied by a resonator in the same direction as the compression. Furthermore, strain-rate cycling was carried out keeping the stress amplitude constant. The stress change due to the strain-rate cycling is $\Delta\tau'$. The tests were conducted at the temperature range of 77 to 178 K. Details of the strain-rate cycling test associated with the oscillation have been reported in the previous papers [23, 24].

The specimens, which were prepared by cleaving out of a block, are the size of about $5 \times 5 \times 15$ mm^3. They were kept at 973 K for 24 h and were cooled to room temperature at a rate of 40 Kh^{-1} in order to reduce dislocation density. Furthermore, they were held at 673 K for 30 min and were followed by water quenching in order to disperse the impurities immediately before the tests.

3. Results and discussion

3.1. Consideration with $m_{\varepsilon=0}$

If the dislocation velocity is controlled by the thermally activated overcoming of the short-range stress field around the I-V dipoles and the dislocation moves forward a distance, L, the dislocation velocity is given by [25]

$$\upsilon = \nu L \exp(-\Delta G/kT) \qquad (2)$$

where ν is the frequency of vibration of a dislocation segment of length, L, ΔG is the change in Gibbs free energy of activation for the dislocation motion, and kT has the usual meaning. The ΔG in Equation 2 is expressed for the Fleischer's model [1] taking account of the Friedel relation [4] as follows [26]

$$\Delta G = F_0 b\{1 - (\tau/\tau_0)^{1/3}\}^2 \qquad (3)$$

where F_0 is the force acted on the dislocation at the temperature of 0 K, b is the magnitude of the Burgers vector, and τ_0 is the effective shear stress due to the impurities at 0 K. This model is termed the F-F in this paper. Substituting Equation 3 in Equation 2 gives

$$\upsilon = \nu L \exp[-F_0 b\{1 - (\tau/\tau_0)^{1/3}\}^2/(kT)] \qquad (4)$$

Natural logarithmic differentiation of Equation 1 with respect to the shear stress gives

$$m^* = \Delta \ln \upsilon/\Delta \ln \tau = \tau(\Delta \ln \upsilon/\Delta\tau) \qquad (5)$$

The $(\Delta \ln \upsilon/\Delta\tau)$ in Equation 5 can be obtained from Equation 4 as follows

$$\Delta \ln \upsilon/\Delta\tau = 2F_0 b/(3kT\tau_0)\{(\tau/\tau_0)^{-2/3} - (\tau/\tau_0)^{-1/3}\} \qquad (6)$$

Substituting Equation 6 in Equation 5, the m^* for the F-F is expressed by

$$m^* = 2F_0 b\{(\tau/\tau_0)^{1/3} - (\tau/\tau_0)^{2/3}\}/(3kT) \qquad (7)$$

Furthermore, substituting the following relation [26] between effective shear stress and temperature into Equation 7, we can evaluate the m^* for the F-F.

$$(\tau/\tau_0)^{1/3} = 1 - (T/T_c)^{1/2} \qquad (8)$$

Namely,

$$m^* = 2F_0 b\{(T_c/T)^{1/2} - 1\}/(3kT_c) \qquad (9)$$

where T_c is the critical temperature at which τ_{p1} is zero. T_c is independent of an impurity concentration [23, 24]. τ_{p1} is considered to represent the effective shear stress due to only one type of impurities when a dislocation moves forward with the help of oscillation [23, 24]. The τ_{p1} is obtained on the basis of the strain-rate sensitivity, λ, versus stress decrement curves due to oscillation [23, 24]. The F_0 in Equation 9 is determined by [26]

$$F_0 = \left(2L_0^2 E\right)^{1/3}(b\tau_{p0})^{2/3} \qquad (10)$$

where L_0 is the average spacing of impurities on the slip plane, E is the line tension of the dislocations, and τ_{p0} is the value of τ_{p1} at 0 K. The line tension of the dislocations is calculated by μb^2. The shear modulus, μ, for [110] direction is assumed to be 1.01×10^{10} Pa at 0 K [27]. The average spacing of impurities on the slip plane has been given by [1, 28]

$$L_0 = b/(4c/3)^{1/2} \qquad (11)$$

where c is the concentration of impurities and is determined to be 7.9 p.p.m. by dielectric loss measurement.

On the other hand, the ΔG in Equation 2 is expressed for the Fleischer's model as follows [1]

$$\Delta G = F_0 b\{1 - (\tau/\tau_0)^{1/2}\}^2 \qquad (12)$$

Combination of Equations 2 and 12 yields

$$\upsilon = \nu L \exp[-F_0 b\{1 - (\tau/\tau_0)^{1/2}\}^2/(kT)] \qquad (13)$$

Natural logarithmic differentiation of Equation 13 with respect to $\ln\tau$ gives [1]

$$m^* = F_0 b\{(\tau/\tau_0)^{1/2} - (\tau/\tau_0)\}/(kT) \qquad (14)$$

and the relative formula of effective shear stress and temperature for the Fleischer's model is given by [1]

$$(\tau/\tau_0)^{1/2} = 1 - (T/T_c)^{1/2} \qquad (15)$$

The Fleischer's model has been widely used for the interaction between a dislocation and an impurity in ionic crystals doped with divalent cations [1, 2, 29, 30]. Substituting Equation 15 into Equation 14, we find

$$m^* = F_0 b\{(T_c/T)^{1/2} - 1\}/(kT_c) \qquad (16)$$

where F_0 is determined by

$$F_0 = \tau_{p0} L b \qquad (17)$$

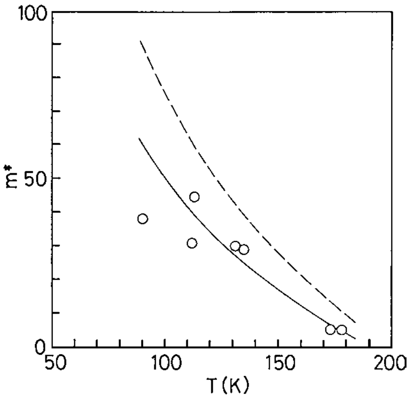

Figure 1 Relationship between the temperature and the dislocation velocity-effective stress exponent for KCl:Mg^{2+} (0.035 mol% in the melt) at the two models: (- - - -) the F-F and (——) the Fleischer's model. Open circles represent the $m_{\varepsilon=0}$ for the specimen.

The m^* of Equation 16 corresponds to that for the Fleischer's model. Temperature versus m^* curves calculated from Equations 9 and 16 are represented by a dashed and a solid lines in Fig. 1. Then, the values of T_c and τ_{p0} for the F-F are obtained from Fig. 2a and those for the Fleischer's model from Fig. 2b, which are re-tabulated in Table I. The two curves show that m^* increase with decreasing temperature. The m^* value for the F-F is greater than that for the Fleischer's model at a given temperature. The difference between m^* values for the two models also increases with decreasing temperature, which ranges 3.9 to 28.6 at the temperature of 190 to 90 K.

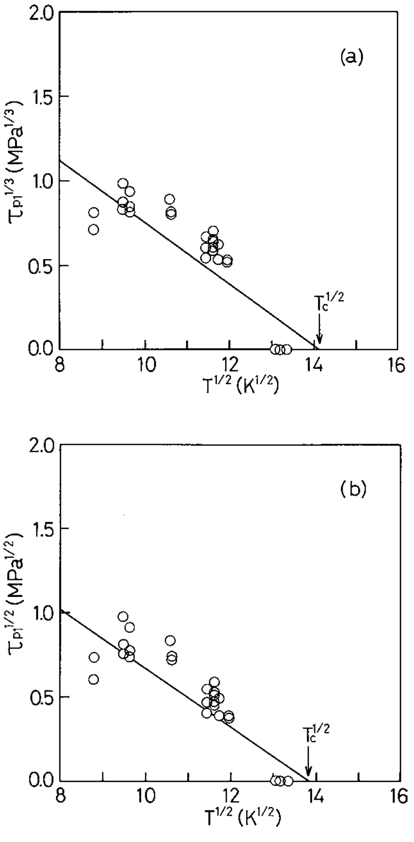

Figure 2 Linear plots of the effective shear stress and the temperature for KCl:Mg^{2+} (0.035 mol% in the melt) at the two models: (a) the F-F and (b) the Fleischer's model.

TABLE I Values of T_c and τ_{p0} for KCl:Mg^{2+} (0.035 mol% in the melt) at the two models

Model	T_c (K)	τ_{p0} (MPa)
F-F	199 [31]	17.91 [31]
Fleischer	191 [32]	5.64 [33]

According to the theory by Nix *et al.* [34], the value of m^* is identical with m when τ_i is considerably small in comparison with τ_a. m is a dislocation velocity-applied stress exponent. τ_i and τ_a are internal stresses and applied stress respectively. The work hardening in both stage I and stage II of plastic deformation region was largely due to internal stress components in LiF [35] and NaCl [15, 35, 36] single crystals. Then, the effective stress due to a small amount of impurity was almost constant independently of strain during the plastic deformation. Therefore, the value of m^* seems to almost agree with $m_{\varepsilon=0}$ (m value at zero strain) for a given material and temperature. Furthermore, the influence of internal stresses on dislocation velocity in zinc could be neglected at the applied shear stress below about 0.01 MPa [37]. This also implies that the value of m^* $(=\Delta \ln v/\Delta \ln \tau)$ corresponds to m $(=\Delta \ln v/\Delta \ln(\tau + \tau_i))$ at extremely small strain and a given temperature. Johnston and Stein [38] have proposed that m^* can be determined by extrapolating m to zero strain for LiF single crystal. Evans and Pratt [36] showed that m^* compares well with m at zero strain in CaF_2 single crystals. In addition, the values of m^* and $m_{\varepsilon=0}$ for NaCl single crystals at 244 K were 13 ± 2 and 16 ± 2 respectively [36]. The $m_{\varepsilon=0}$ was independent of the strain-rate increments: $\dot{\varepsilon}_1/\dot{\varepsilon}_2 = 2, 4, 10, 40, 100, 400,$ and 1000 [36]. $m_{\varepsilon=0}$ for alkali halide was considered to be m^*, since the thermal components of flow stress are independent of mobile dislocation density at low concentration of impurities and near zero strain, where the thermally activated overcoming of impurities by a dislocation is dominant [39]. Deformation behavior has been investigated with $m_{\varepsilon=0}$ so far [14, 36, 40–42]. Using $m_{\varepsilon=0}$, reasonable value of effective stress and hardening mechanism were found for zone-refined iron [40] and potassium contained Na^+ (25 p.p.m.) [41] during the deformation.

Fig. 3 shows m versus shear strain curve for KCl:Mg^{2+} (0.035 mol%). The m values are obtained here by strain-rate cycling tests without applying the oscillation. Through the m value extrapolated to zero strain in Fig. 3, therefore m^* seems to be about 29 at 135 K on the assumption that internal stresses and mobile dislocation density are unchanged during the change in strain rate of the specimen, because m^* is defined as [38]

$$(\Delta \ln \dot{\varepsilon}/\Delta \ln \tau)_T = m^* + (\Delta \ln \rho/\Delta \ln \tau)_T \qquad (18)$$

where ρ is the density of mobile dislocations. The $(\Delta \ln \dot{\varepsilon}/\Delta \ln \tau)_T$ in this study is given by $(1.609/\Delta \ln \tau)$. Open circles in Fig. 1 correspond to the dependence of temperature and the $m_{\varepsilon=0}$ for KCl:Mg^{2+}

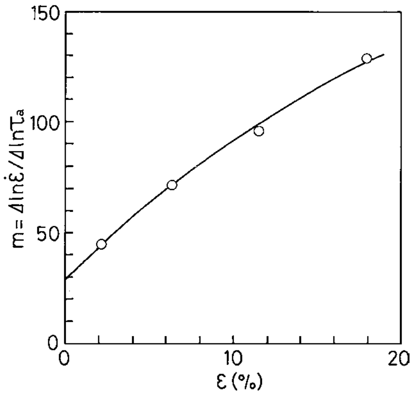

Figure 3 Variation of m with shear strain for KCl:Mg^{2+} (0.035 mol% in the melt) at 135 K.

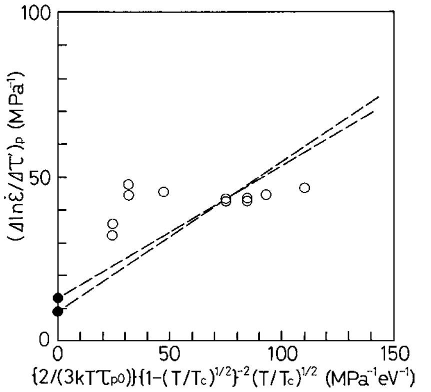

Figure 4 Linear plots of Equation 20 for KCl:Mg^{2+} (0.035 mol% in the melt). (○): $(\Delta \ln \dot\varepsilon/\Delta\tau')_p$; (●): $\partial \ln \dot\varepsilon_0/\partial\tau$.

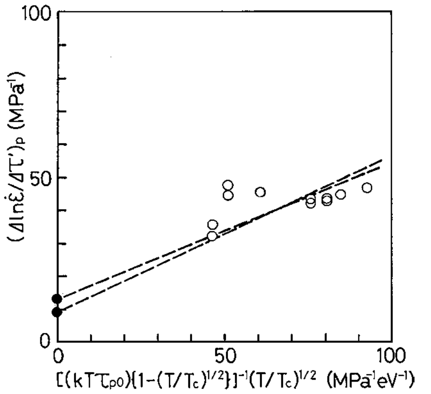

Figure 5 Linear plots of Equation 22 for KCl:Mg^{2+} (0.035 mol% in the melt) [33]. (○): $(\Delta \ln \dot\varepsilon/\Delta\tau')_p$. (●): $\partial \ln \dot\varepsilon_0/\partial\tau$.

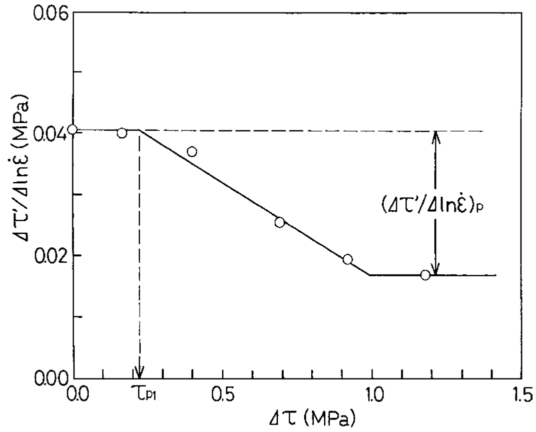

Figure 6 Relationship between the strain-rate sensitivity and the stress decrement for KCl:Mg^{2+} (0.035 mol% in the melt) at 131 K and ε = 10%.

(0.035 mol%). The open circles tend to increase with decreasing temperature and approach to solid line. This suggests that the interaction between a dislocation and the impurity in the specimen can be approximated to the Fleischer's model rather than to the F-F. That is, the F-F is considered to be unsuitable for the specimen. Consequently, it may be deduced that the Friedel relation is not appropriate for the interaction between a dislocation and the impurity in the specimen within the temperature range.

3.2. Consideration with $\tau_{p1}(\Delta\ln\dot\varepsilon/\Delta\tau')_p$

The m^* for the F-F may be also given by Equation 7 transformed into

$$m^* = 2\Delta G_0\{(\tau_{p1}/\tau_{p0})^{1/3} - (\tau_{p1}/\tau_{p0})^{2/3}\}/(3kT) \qquad (19)$$

where ΔG_0 is the Gibbs free energy for the breakaway of the dislocation from the impurity in the absence of an applied stress and is obtained from the following equation [26]:

$$\partial\ln\dot\varepsilon/\partial\tau = \{2\Delta G_0/(3kT\tau_{p0})\}\{1-(T/T_c)^{1/2}\}^{-2} \times (T/T_c)^{1/2} + \partial\ln\dot\varepsilon_0/\partial\tau \qquad (20)$$

The calculations of Equation 20 are shown by open circles in Fig. 4. As for the Fleischer's model, the m^* may be given by the following equation in accordance with Equation 14. Namely,

$$m^* = \Delta G_0\{(\tau_{p1}/\tau_{p0})^{1/2} - (\tau_{p1}/\tau_{p0})\}/(kT) \qquad (21)$$

and ΔG_0 in Equation 21 is obtained from [33]

$$\partial\ln\dot\varepsilon/\partial\tau = \{\Delta G_0/(kT\tau_{p0})\}\{1-(T/T_c)^{1/2}\}^{-1} \times (T/T_c)^{1/2} + \partial\ln\dot\varepsilon_o/\partial\tau \qquad (22)$$

Open circles in Fig. 5 show the results of Equation 22. The $\partial\ln\dot\varepsilon/\partial\tau$ in Equations 20 and 22 is represented by $(\Delta\ln\dot\varepsilon/\Delta\tau')_p$. The $(\Delta\ln\dot\varepsilon/\Delta\tau')_p$, which is given by the difference between λ at first plateau place and at second one on the relative curve of λ and stress decrement, was assumed to be the λ due to the impurities [32, 43–45]. Fig. 6 shows the typical variation of λ with $\Delta\tau$ for the specimen. The $\Delta\tau$, which is an axis of abscissa in Fig. 6, is the stress drop due to superposition of the ultrasonic oscillatory stress. Accordingly, $\partial\ln\dot\varepsilon_0/\partial\tau$ in Equations 20 and 22 could be evaluated from $\Delta\ln\dot\varepsilon_0/\Delta\tau'$ [33], which is represented by the solid circles in Figs 4 and 5. On the basis of the slope of dashed lines in Figs 4 and 5, the ΔG_0 for the specimen at the F-F was estimated to be within the

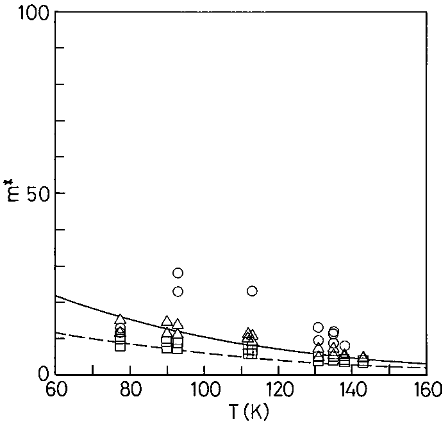

Figure 7 Relationship between the temperature and the dislocation velocity-effective stress exponent for KCl:Mg^{2+} (0.035 mol% in the melt) at the two models: (□) the F-F and (△) the Fleischer's model. Open circles represent $\tau_{\rm p1}(\Delta \ln \dot{\varepsilon}/\Delta\tau')_{\rm p}$ for the specimen. (----) and (——) are calculated by Equations 9 and 16, respectively.

range of 0.40 to 0.46 eV and that at the Fleischer's model 0.42 to 0.48 eV [33]. The m^* values calculated from Equations 19 and 21 are denoted by open squares and triangles in Fig. 7. Dashed and solid curves correspond to the results of Equations 9 and 16, being already shown in Fig. 1. These open symbols tend to increase slightly with decreasing temperature. Furthermore, m^* may be analyzed in terms of λ versus stress decrement curve due to oscillation as follows

$$m^* = \tau_{\rm p1}(\Delta \ln \dot{\varepsilon}/\Delta\tau')_{\rm p} \tag{23}$$

This m^* is indicated by open circles in Fig. 7. The values of m^* in Equations 19, 21, and 23 are examined on the assumption that Equation 5 is also valid for the case of applying the ultrasonic oscillatory stress during plastic deformation. As can be seen from Fig. 7, the open circles are closer to m^* for the Fleischer's model in contrast to that for the F-F, though the difference of the two (i.e., open circles and triangles) is great at a given temperature. This means that the interaction between a dislocation and the impurity in the specimen can be fitted to the Fleischer's model rather than to the F-F. Resulting in the same description as preceding section, it is considered that the Friedel relation is not appropriate for the specimen within the temperature range.

4. Conclusion

1. Temperature versus m^* for the F-F can be examined from Equation 9 and that for the Fleischer's model from Equation 16. Fig. 1 shows the results for KCl:Mg^{2+} (0.035 mol% in the melt). The m^* values for the two models increase with decreasing temperature. Assuming that internal stresses and mobile dislocation density in the specimen are unchanged during the change in strain rate, it is considered that m^* can be expressed by $m_{\varepsilon=0}$. Observing open circles in Fig. 1, the $m_{\varepsilon=0}$ tends to increase with decreasing temperature and further approaches to the m^* for the Fleischer's model rather than that for the F-F at the temperature.

2. The m^* values for the F-F and for the Fleischer's model are calculated also from Equations 19 and 21 respectively. Then the m^* values proposed by Equation 23 are closer to the m^* for the Fleischer's model, compared with that for the F-F, as shown in Fig. 7. The m^* of Equation 23 is obtained in terms of λ versus stress decrement curve due to oscillation. The discussion in this study is progressed on the assumption that Equation 5 can be also applied to the case of applying the ultrasonic oscillatory stress during plastic deformation.

Figs 1 and 7 imply that the Fleischer's model is suitable to the interaction between a dislocation and the impurity in the specimen, whereas the F-F is unsuitable to it. This leads to the estimation that the Friedel relation is inappropriate to that in the specimen within the temperature range.

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