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Orientation and temperature dependence of the flow stress in the intermetallic compound Ni_3Ge single crystals

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Tensile tests on Ni_3Ge single crystals were carried out to make clear the mechanism of the positive temperature dependence of the CRSS and the work-hardening rate as a function of the tensile axis orientation below room temperature. Both the CRSS (τ) and the maximum work hardening rate (θ_M) show positive temperature dependence even below room temperature. The increments of τ and θ_M are satisfactorily expressed by the Schmid factor ratio of the cube cross slip system to the primary octahedral one, $N = (010)\ [\bar{1}01]/(111)\ [\bar{1}01]$. Then, the positive temperature dependence of both the CRSS and the maximum work-hardening rate is thought to be governed by two mechanisms. One arises from the Kear–Wilsdorf mechanism, which depends on the orientation. The other seems to arise from the orientation-independent factor, although it is obscure at present.

1. Introduction

It is known that the yield stress of the $L1_2$ intermetallic compounds (Ni_3Al [1–6], Ni_3Ge [7, 8], Ni_3Ga [9] and so on) with high APB (antiphase domain boundary) energies increases remarkably with increasing temperature, reaching a peak value at an elevated temperature T_p, depending on alloying additions, deviations from stoichiometry and the orientation of the tensile axis. As the positive temperature dependence is a common phenomenon in $L1_2$ compounds, it may be controlled by the same mechanism.

The main experimental knowledge of the deformation behaviour in $L1_2$ compounds is summarized as follows. (i) The macroscopic yield (10^{-3} strain) stress shows positive temperature dependence, while the microyield (10^{-6} strain) stress is almost constant over a wide range of temperature [3, 10]. Furthermore, screw dislocations are mainly observed at the positive temperature-dependence range by transmission electron microscopy [7, 8, 11]. (ii) The operative slip planes are the $\{111\}$ planes below peak temperature, T_p, (at the positive temperature-dependence region), but they become the $\{100\}$ planes at elevated temperature above T_p [7, 8, 11–13]. (iii) The critical resolved shear stress (CRSS) for the primary octahedral slip system $(111)\ [\bar{1}01]$ is orientation dependent, the larger the stress component on the (010) cube cross slip plane, the higher the CRSS [7–9, 11].

At present the positive temperature dependence of the yield stress in $L1_2$ compounds is said to be caused by the decrease in mobility of screw dislocations on the $\{111\}$ planes as the deformation temperature increases based on both (i) and (ii). Furthermore, the decrease in mobility of screw dislocations may be related to the cross slip of superdislocations onto (010) planes to reduce the total energy of dislocations (so called the Kear–Wilsdorf mechanism [14]), and the cross slipped parts act as dragging points for the dislocation motion on the $\{111\}$ plane based on (iii). However, there is little experimental evidence for supporting the cube cross slip mechanism.

Recently, the authors clarified that the positive temperature dependence of the CRSS in $Ni_3(Al \cdot Ti)$ single crystals is thought to be governed by two mechanisms [15]. That is, one arises from the Kear–Wilsdorf mechanism, which depends on the orientation. The other seems to arise from the orientation-independent factor, although it is obscure at present.

The compression tests on Ni_3Ge single crystals were performed by Pak, Saburi and Nenno [7, 8]. According to them, though the positive temperature dependence of the CRSS in Ni_3Ge was qualitatively explained by the above-mentioned cube cross slip mechanism, no direct evidence was obtained. Therefore, it is not sufficiently clear whether the decrease in the mobility of screw dislocations resulted from the dragging due to the cube cross slip or not.

On the other hand, it was reported that the work hardening rate in Ni_3Al single crystals also shows the positive temperature dependence [2]. The positive temperature dependence of the work-hardening rate and the mechanism of it in $L1_2$ intermetallic compounds are little investigated. Recently, the present authors investigated into the mechanism of the positive temperature dependence of the work-hardening rate in $Ni_3(Al \cdot Ti)$ single crystals and have concluded that it is governed by the Kear–Wilsdorf mechanism alone [15].

However, it is not known whether the positive temperature dependence of the work-hardening rate in other $L1_2$ intermetallic compounds is also controlled by the Kear–Wilsdorf mechanism alone. A further systematic investigation on the orientation dependence of the deformation behaviour is necessary for elucidating the mechanism of the positive temperature dependence of the yield stress and the work-hardening rate in $L1_2$ intermetallic compounds. In the present paper, the results of an experimental investigation on the orientation dependence of the yield stress and the work-hardening rate in Ni_3Ge single crystals below room temperature (the region revealing the positive temperature dependence) are reported.

2. Experimental procedure

Nickel (99.9%) and germanium (99.9999%) were used for the preparation of off-stoichiometric Ni_3Ge(23.2 at.% Ge) by melting in a high purity alumina crucible and casting into an iron mould under a purified argon atmosphere. A single crystal of Ni_3Ge, 150 mm in length and 25 mm in diameter, was grown from a polycrystalline ingot in a high purity alumina mould by the Bridgman technique. The growth chamber was filled with high purity argon gas and was moved down at a rate of $60\,\mathrm{mm\,h^{-1}}$. The single crystal was homogenized for 50 h at 1323 K in a vacuum of 1.33 mPa. Tensile specimens were cut from the one single crystal, excluding both ends of it, using an electronic spark cutting machine. The specimens had rectangular cross-sections and a gauge size of approximately 1 mm x 1 mm x 12 mm. Seven different orientations were chosen on the boundary line of the standard stereographic triangle as shown in Fig. 1, where the [A] orientation is located on the $[001]-[\bar{1}11]$ symmetry line and is 11° apart from the $[\bar{1}11]$ to $[001]$ orientation. The orientation of specimens was determined by the Laue back-reflection method, and their orientations were shown to be within 2° of the expected one.

Table I shows both N and M values for each specimen, where N is the Schmid factor ratio of $(010)\ [\bar{1}01]$ slip (cube cross slip) to $(111)\ [\bar{1}01]$ slip and is considered to be a measure of the resolved shear stress promoting cross slip from (111) onto (010) planes. On the other hand, M is the Schmid factor ratio of $(\bar{1}\bar{1}1)\ [011]$ slip (conju-

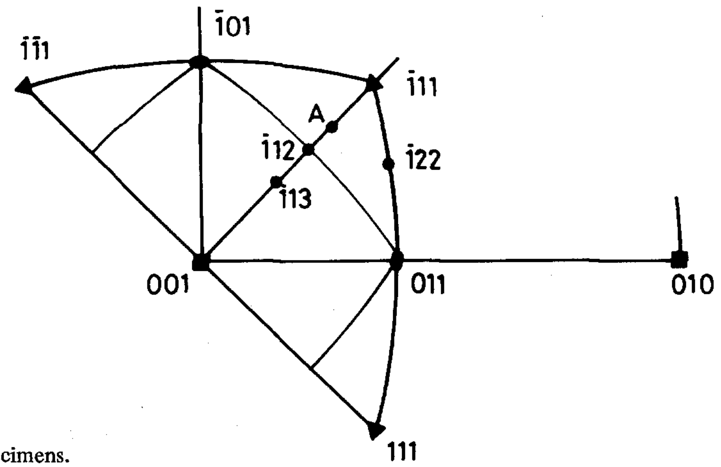

Figure 1 The orientation of the tensile specimens.

TABLE I Schmid factor ratios N and M for each specimen

Tensile axis	N, (010) $[\bar{1}01]$/ (111) $[\bar{1}01]$	M, $(\bar{1}\bar{1}1)$ [011]/ (111) $[\bar{1}01]$
[001]	0	1.0
$[\bar{1}13]$	0.58	1.0
$[\bar{1}12]$	0.87	1.0
[A]	1.11	1.0
$[\bar{1}11]$	1.73	1.0
$[\bar{1}22]$	1.15	0.42
[011]	0.87	0

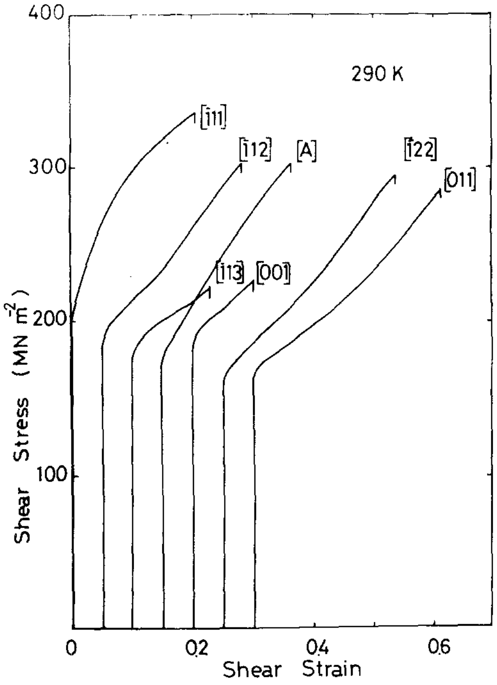

Figure 2 Shear stress versus shear strain curves at 290 K.

gate slip) to (111) $[\bar{1}01]$ slip and is considered to be a measure of the resolved shear stress promoting conjugate slip. After spark cutting, the damaged surface layer of the specimens was removed by electrolytic polishing (25% HClO$_4$ and 75% C$_2$H$_5$OH) and then the specimens were annealed again for 24 h at 1327 K. Finally, all specimens were electrolytically polished.

Tensile tests were carried out in an Instron-type machine. The specimens were pulled at an initial strain rate of 1.3×10^{-3} sec^{-1}.

3. Experimental results

3.1. Stress–strain curves

Fig. 2 shows the resolved shear stress versus shear strain curves at 290 K assuming that slip occurs only on the primary plane in the [011] and $[\bar{1}22]$ crystals, on both primary and conjugate glide planes in the $[\bar{1}13]$, $[\bar{1}12]$ and [A] crystals, on three glide planes in the $[\bar{1}11]$ crystals, and four glide planes in the [001] crystals. These assumptions were confirmed to be reasonable by the observations of the slip bands, cross-sectional changes and rotations of tensile axes.

The $[\bar{1}11]$ orientation gives only parabolic hardening at every temperature, while the others show easy and/or linear hardening and do not clearly show parabolic hardening with the exception of the [A] orientation at 290 K. The absence of the parabolic work hardening is characteristic of the deformation behaviour in Ni$_3$Ge single crystals. Similar curves were obtained for other temperatures. The elongation and the ultimate tensile

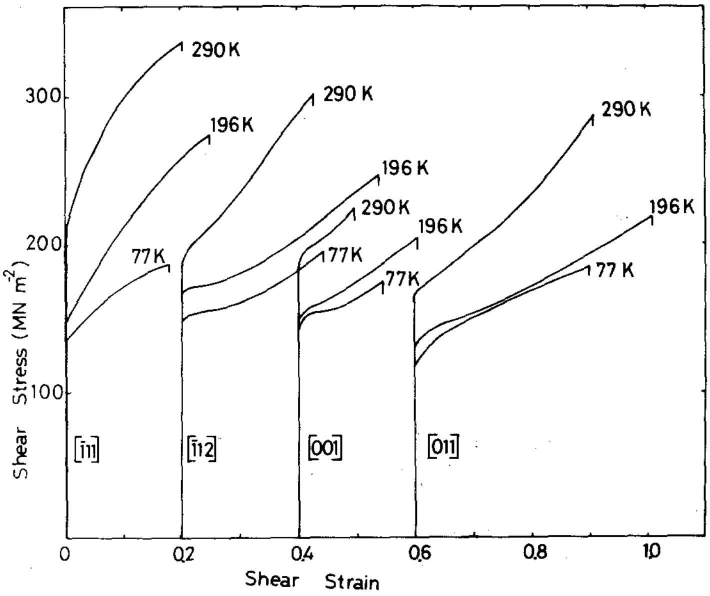

Figure 3 Temperature dependence of the shear stress versus shear strain curves at typical orientations.

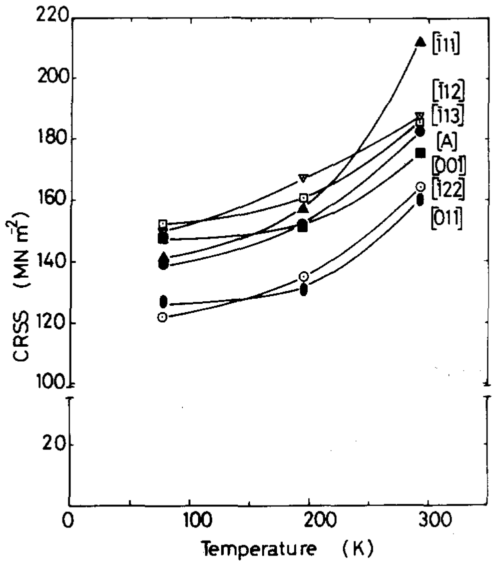

Figure 4 Temperature and orientation dependence of the CRSS.

strength in [001] and [$\bar{1}$13] orientations are always slightly lower than those in the other orientations.

Fig. 3 shows the temperature dependence of the shear stress versus shear strain curves for typical orientations. As can be seen in the figure, the yield stress, work-hardening rate and ultimate tensile stress (approximately equal to the fracture stress) increase with increasing temperature.

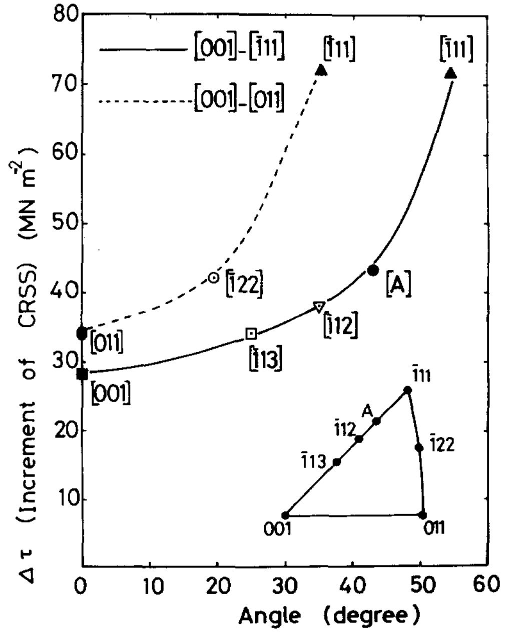

Figure 5 The increment of the CRSS by temperature increase from 77 K to 290 K as a function of angles from the [001] or [011] orientations.

3.2. Temperature and orientation dependence of the CRSS

Fig. 4 shows the temperature and orientation dependence of the critical resolved shear stress (CRSS) for seven crystals below room temperature. It is seen that the CRSS of Ni_3Ge similarly increases with increasing temperature as in the case of $Ni_3(Al \cdot Ti)$ [15]. The increment of the CRSS becomes more prominent especially above about 200 K. This positive temperature dependence may persist up to high temperatures and form the peak of the yield stress [7, 8], depending on the orientation.

Fig. 5 shows the increment of the CRSS ($\Delta\tau$) with the increase of temperature from 77 to 290 K as a function of the angles either from the [001] or [011] orientations. The solid line expresses the change of $\Delta\tau$ with angle from the [001] orientation to the [$\bar{1}$11] one along the [001]–[$\bar{1}$11] symmetry line, while the dotted line shows that from the [011] orientation to the [$\bar{1}$11] one along the [011]–[$\bar{1}$11] line. As can be seen in this figure, the increment of CRSS becomes more prominent as the orientation of the tensile axis approaches the [$\bar{1}$11] orientation. These tendencies are qualitatively in agreement with those of previous works [7, 8, 15].

3.3. Temperature and orientation dependence of the work-hardening rate

Fig. 6 shows the temperature and orientation dependence of the maximum work-hardening rate (θ_M) obtained from the slope of the shear stress versus shear strain curves. The θ_M value also shows positive temperature dependence similar to that of

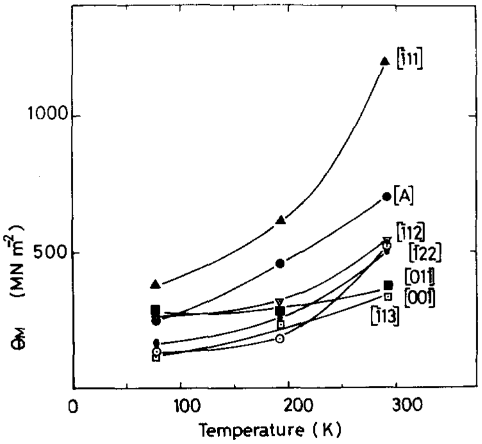

Figure 6 Temperature and orientation dependence of the maximum work-hardening rate obtained from the slope of the shear stress versus shear strain curves in Fig. 2.

CRSS. It is noticed that the θ_M value for the [001] orientation of Ni_3Ge single crystals also shows the positive temperature dependence, while that of $Ni_3(Al \cdot Ti)$ single crystals does not show such a behaviour [15]. The increment of θ_M becomes more prominent with increasing temperatures, especially above about 200 K as can be seen in this figure.

Fig. 7 shows the increment of θ_M with an increase of temperature from 77 to 290 K as a function of angle either from the [001] or the [011] orientations. The increment of θ_M is also more prominent as the orientation of the tensile axis approaches the $[\bar{1}11]$ orientation, similar to the case of CRSS.

4. Discussion

4.1. The positive temperature dependence of the CRSS

The orientation dependence of the increment of the CRSS ($\Delta\tau$ in Fig. 5) strongly suggests that the positive temperature dependence may closely connect with the cross slip of superdislocations from the {111} planes onto the {100} planes, because the cube cross slip process becomes easier on approaching the $[\bar{1}11]$ direction.

It has recently been pointed out that the CRSS τ in the $L1_2$ intermetallic compounds may have two components; a temperature-independent one which dominates at low temperatures and a temperature-dependent one which dominates at higher temperatures [16]. Therefore, the CRSS $\tau_{(T)}$ for the $(111)\,[\bar{1}01]$ slip system at T K is given by the sum τ_0, temperature-independent stress and τ^*, temperature-dependent one.

$$\tau_{(T)} = \tau_0 + \tau^* \qquad (1)$$

Where τ_0 will be determined by the CRSS at low temperature where τ is mostly independent of temperature. The positive temperature dependence results from only the second term τ^*, which depends on the orientation of the tensile axes and the test temperature. The temperature dependence of τ at 77 K is so small that τ at 77 K, as a first approximation, can be regarded as τ_0. Then, the difference in τ between 77 K and 290 K (namely, $\Delta\tau$ in Fig. 5) is equal to τ^* at 290 K.

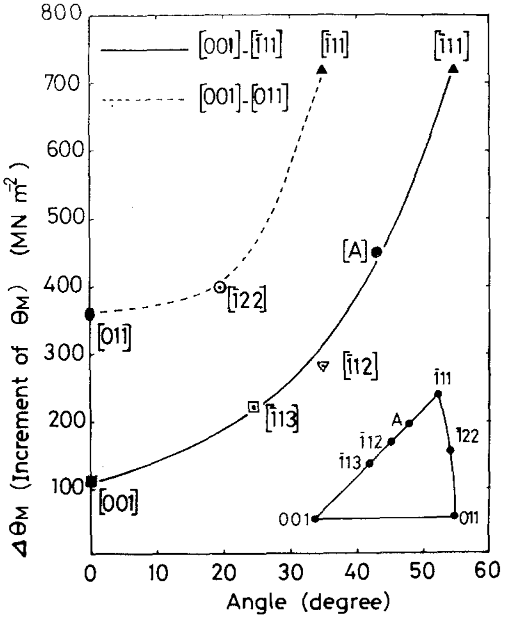

Figure 7 The increment of the maximum work-hardening rate by the temperature increase from 77 K to 290 K as a function of angle from the [001] or [011] orientation.

On the other hand, it is well known that a superdislocation in ordered alloys consists of a pair of two component dislocations bound by the APB ribbon. According to Flinn [17], APB energy in the $L1_2$ structure takes a minimum value on the {100} plane. Hence, superdislocations on the primary octahedral slip plane {111} have a tendency to cross slip onto the {100} to reduce the total energy of dislocations. This process is known as the Kear–Wilsdorf mechanism [14], and depends on the Schmid factor ratio N, $(010)\,[\bar{1}01]/(111)\,[\bar{1}01]$, because N is a measure of the resolved shear stress promoting cross slip from (111) onto (010) planes.

Fig. 8 shows the relation between τ^* and N value at 290 K. The τ^* increases obviously with increasing N values as seen in this figure. Therefore, it can be concluded that the positive temperature dependence of the CRSS is clearly controlled by the Kear–Wilsdorf mechanism, namely, the screw superdislocations on the {111} plane locally cross slip onto the {100} plane with a thermally-activated process. The resistance to dislocation motion is so large on the {100} planes that the cross-slipped segments prevent the subsequent motion of the remaining parts of the dislocations on the (111) $[\bar{1}01]$ slip system. The increment of the CRSS on the {111} ⟨110⟩ slip systems increases with increasing the number of cube cross slip parts. As the probability of the cube cross slip increases with increasing temperatures and N values, the positive temperature dependence of the CRSS tends to depend upon the orientation. Thus, the temperature and the orientation dependences of

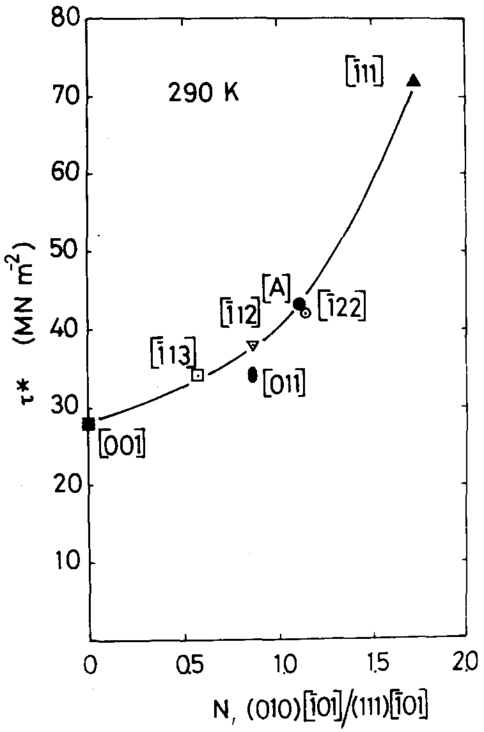

Figure 8 The relation between τ^* at 290 K and N value which expresses the operation of the cube cross slip process.

the CRSS in Ni_3Ge single crystals below room temperature (Figs. 4 and 5) are qualitatively explained based on the Kear–Wilsdorf mechanism. However, the positive temperature dependence of the CRSS is not controlled only by the Kear–Wilsdorf mechanism. If it were, the increment of the CRSS must be nullified when the N value is zero. However, τ^* did not become zero at $N = 0$ as can be seen in Fig. 8. The positive temperature dependence of τ in the [001] orientation has also been reported for $Ni_3(Al \cdot Ti)$ single crystals over a wide range of temperatures [4, 15]. These facts imply that even if the stress component onto (010) is null, τ^* in the [001] orientation is invariably positive. Consequently, another factor which does not relate to the Kear–Wilsdorf mechanism, namely which is independent of orientation, should be taken into account. Although it is vague at present how this factor is governed, it is characterized by its dependence, not on orientation, but on temperature. As suggested by Mulford and Pope [10], and Liang and Pope [16], this factor may result from the relaxation of the core structure in screw dislocations forming a sessile configuration. Further investigation is necessary in this field. Thus, the positive temperature dependence of the CRSS in Ni_3Ge single crystals is considered to be introduced by two mechanisms. The fundamental one may arise from the cross slipping of screw superdislocations onto (010) planes (the so-called Kear–Wilsdorf mechanism) leading to the dragging of dislocations. Another one seems to arise from the orientation-independent factor, though it is obscure at present. These conclusions in the present work strongly support the previous results on $Ni_3(Al \cdot Ti)$ single crystals, namely, the positive temperature dependence of CRSS is thought to be governed by two mechanisms [15].

4.2. The positive temperature dependence of the work-hardening rate

It has been found in the present work that the maximum work-hardening rate of Ni_3Ge single crystals also shows positive temperature dependence as seen in Fig. 6. Generally, it is known that the work-hardening rate in ordered alloys is higher than that in disordered ones [18]. The positive temperature dependence of the work-hardening rate in Ni_3Ge seems to result from ordering effects.

Several theories, based on the existence of the superdislocations, have been put forward to explain the higher work-hardening rate in fully ordered $L1_2$ alloys. Among them, we can find two theories which are attractive for explaining the positive temperature dependence of θ_M in Ni_3Ge.

One theory is the cross slip of screw superdislocations from $\{111\}$ onto $\{100\}$ leading to a sessile configuration as proposed by Kear and Wilsdorf (which was discussed in detail above) [14]. Another one is the formation of APB tubes by non-aligned jogs on superdislocations as proposed by Vidoz and Brown [19]. Thus, the Kear–Wilsdorf mechanism is commonly proposed as the mechanism which may explain the positive temperature dependences of both the CRSS and the work-hardening rate.

As the temperature dependence of θ_M at 77 K is very small (as seen in Fig. 6), $\theta_{M(T)}$ at T K is expressed in a similar manner as the case of the CRSS,

$$\theta_{M(T)} = \theta_{M(0)} + \theta_M^* \qquad (2)$$

where, $\theta_{M(0)}$ is the temperature-independent term and approximately equals to $\theta_{M(77\,K)}$. The positive temperature dependence results from the second term, θ_M^*. The difference in θ_M between 77 K and

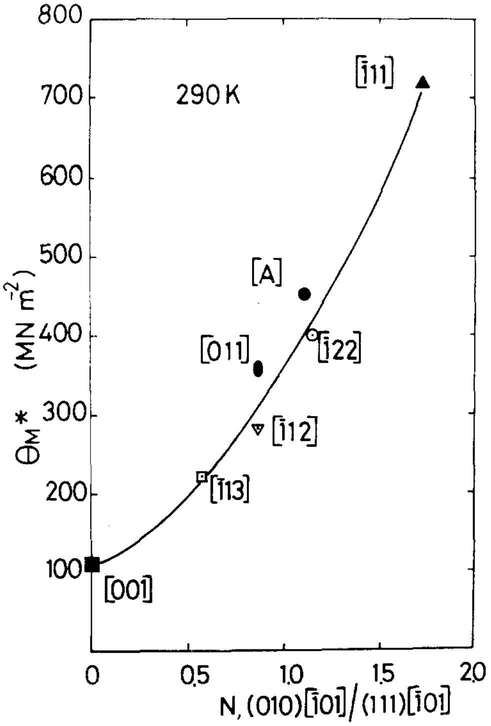

Figure 9 The relation between θ_M^* at 290 K and N value which expresses the operation of the cube cross slip process.

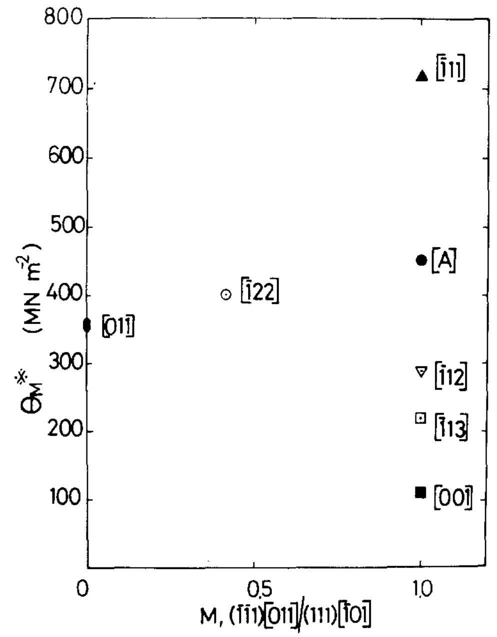

Figure 10 The relation between θ_M^* at 290 K and M value which expresses the operation of the APB tube mechanism.

290 K is regarded as θ_M^* of 290 K (equals $\Delta\theta_M$ in Fig. 7).

Fig. 9 shows the relationship between θ_M^* at 290 K and the N value. θ_M^* increases obviously with increasing N values as seen in this figure. The parabola does not pass through the origin as seen in Fig. 9, thus the positive temperature dependence of the θ_M in Ni_3Ge is considered to be produced by two mechanisms. The fundamental one may arise from the Kear–Wilsdorf mechanism. Another one seems to arise from the orientation-independent factor, though it is obscure at present.

On the other hand, it has been shown that the positive temperature dependence of the θ_M in $Ni_3(Al \cdot Ti)$ single crystals is governed only by the Kear–Wilsdorf mechanism, because the parabola passes through the origin [15]. Therefore, there is a difference in work-hardening behaviours between Ni_3Ge and $Ni_3(Al \cdot Ti)$ single crystals. However, in both cases it is clear that the Kear–Wilsdorf mechanism plays an important role in the work-hardening behaviours of the $L1_2$ intermetallic compounds.

Fig. 10 shows a plot of θ_M^* at 290 K versus M values. No clear relationships can be recognized between θ_M^* and M values as seen in this figure. Therefore, the positive temperature dependence of θ_M cannot be related to the APB tube formation, because the M value is a measure of the resolved shear stress promoting conjugate slip. To produce APB tubes, there must be intersections between the primary and the moving secondary dislocations.

5. Summary and conclusion

Tensile tests on Ni_3Ge single crystals were carried out to elucidate the mechanism of the positive temperature dependence of the CRSS and the work-hardening rate as a function of the tensile axis orientation below room temperature. The main results obtained are summarized as follows.

Both the CRSS (τ) and the maximum work-hardening rate (θ_M) show positive temperature dependence even below room temperature. The increments of τ and θ_M depend on the temperature and the tensile axis orientation, and become more prominent with increasing temperature and with approaching the $[\bar{1}11]$ orientation.

The increments of τ and θ_M are satisfactorily expressed by the Schmid factor ratio of the cube cross slip system to the primary octahedral one, $N = (010)[\bar{1}01]/(111)[\bar{1}01]$. The positive temperature dependence of the CRSS and the maximum work-hardening rate is thought to be governed by two mechanisms. One arises from the Kear–Wilsdorf mechanism, which depends on the orientation. The other seems to arise from the orientation-independent factor, though it is obscure at present.

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