ANALYSIS OF THERMALLY ACTIVATED FORMATION AND BREAKDOWN OF KEAR–WILSDORF BARRIERS IN Ni₃Ge SINGLE CRYSTALS OF VARIOUS ORIENTATIONS

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The orientation dependence of the yield stress in Ni₃Ge single crystals has been examined both theoretically and experimentally. The positive temperature dependence of the yield stress in the lowtemperature region is attributed to formation of Kear–Wilsdorf barriers. The forces driving the formation and breakdown of barriers are calculated within the framework of the Hirsch scheme. A distinctive feature of the model proposed is that the barrier is considered on the screw component of the $a/2[\bar{1}01](111)$ superdislocation in the primary octahedral plane. The major role in barrier formation belongs to anisotropy of elastic moduli, energy of antiphase boundaries in the octahedral plane, shear stresses in the cubic and octahedral planes, and friction-induced stress in the cubic plane. A comparison of predicted values of the driving force of barrier formation and breakdown with experimental values reveals their good agreement. An analysis of the orientation dependence of the driving force of barrier formation in the temperature range T = 77-293 K shows that the dependence $\Delta \tau(T)$ has an extremum for crystals deformed along the [$\bar{1}39$] crystallographic direction, which is confirmed experimentally.

Key words: yield stress, anomaly, thermal hardening, screw dislocations, Kear–Wilsdorf barriers, energy of antiphase boundaries, L1₂ superstructure.

Introduction. An experimental study of the positive temperature dependence of the yield stress τ_c (anomalous) in Ni₃Ge single crystals showed that the orientation of the deformation axis has a pronounced effect on the anomaly [1, 2]. The tendency to cubic-slip activation reduces the peak temperature T_p of the anomaly.

A change in the direction of the deformation axis alters the relative values of shear stresses in the octahedron and in the cubic cross-slip plane, and also the separation of dissociated Shockley partials. Superposition of the above-mentioned factors and anisotropy of elastic moduli are expected to result in a nonmonotonic orientation dependence of the yield-stress anomaly in Ni₃Ge single crystals. Pak et al. [1], however, observed a monotonic growth of the yield stress at room temperature as the deformation axis was deflected from the [0 0 1] direction toward the [1 1 1] direction. No estimates of the driving forces for formation and breakdown of Kear–Wilsdorf (KW) barriers on $a/2[\bar{1} 0 1](1 1 1)$ superdislocations, which define the temperature growth and reduction of the yield stress in Ni₃Ge single crystals (*a* is the lattice parameter), were reported.

The objective of the present study was to analyze the mechanism of formation and breakdown of Kear–Wilsdorf barriers at the moment the leading undissociated superpartial dislocation leaves the plane of its initial location to occupy a position in the cubic cross-slip plane and to theoretically and experimentally examine the orientation dependence of the temperature yield-stress anomaly in Ni₃Ge single crystals on the basis of predicted values of the driving force of formation and breakdown of Kear–Wilsdorf barriers.

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Materials and Experimental Procedure. The procedure used to prepare the samples, perform the mechanical tests of Ni₃Ge single crystals, and examine their dislocation structure (DS) was reported in [2]. The Ni₃Ge single crystals oriented along the [$\bar{1}11$], [$\bar{1}39$], [$\bar{2}34$], [$\bar{4}917$], and [001] crystallographic directions were compressed at a strain rate of $\dot{\varepsilon} = 0.02 \text{ sec}^{-1}$ up to their mechanical failure at T = 77, 293, 523, 673, and 873 K. The deformation at elevated temperatures was performed in a vacuum chamber with a pressure of 10^{-2} mm Hg. The curves of the yield stress $\tau_c(T)$ were plotted in the temperature interval of T = 77–900 K through measured points spaced by 50 K. Each point in the dependences $\tau_c(T)$ was obtained by averaging 3 to 15 measured values of the yield stress.

Temperature and Orientation Dependence of the Yield Stress. Figure 1 shows the yield stress and the rate of change of the yield stress ($\Delta \tau_c / \Delta T$) versus temperature in Ni₃Ge single crystals of different orientations. In crystals oriented along the [$\bar{4}917$], [$\bar{2}34$], and [$\bar{1}11$] directions, the maximum in the curves $\tau_c(T_p)$ was observed close to room temperature. The peak temperatures are indicated by arrows.

A detailed analysis of the curves of the yield stress $\tau_c(T)$ and the flow stress showed that Ni₃Ge single crystals display a pronounced yield-stress anomaly (see Fig. 1a). In the temperature range with a positive anomaly, the yield stress τ_c was found to increase by 4 to 12 times. The orientation of the deformation axis has a pronounced effect on manifestation of the positive temperature dependence. For all the examined orientations, the curves $\tau_c(T)$ display a nonmonotonic behavior in different temperature intervals. As is seen from Fig. 1b and c, the thermalhardening intensity is positive below the peak temperature (T_p) . The temperature T_p depends on the orientation of the deformation axis. The curves $\tau_c(T_p)$ plotted for crystals oriented along the [$\bar{4}917$], [$\bar{2}34$], and [$\bar{1}11$] directions additionally exhibit a weak high-temperature peak whose parameters are almost independent of the orientation of the deformation axis (see Fig. 1a). In the present study, we determined the differences $\Delta \tau(T) = \tau(300) - \tau(77)$ at the initial stage of deformation in samples deformed at T = 77 and 293 K. These differences define the positive growth rate of the yield stress in Ni₃Ge single crystals. In crystals oriented along the [$\bar{2}34$], [$\bar{4}917$], and [$\bar{1}11$] crystallographic directions, these differences coincide with the magnitudes of the flow-stress anomaly at the yield point.

Relation between the Crystallographic Indices of the Deformation Axis and the Parameters that Characterize Mobility of Superdislocations. The temperature growth of the yield stress is caused by variation of mobility of Marcinkowsky screw superdislocations, which represent a pair of $a/2[\bar{1}01](111)$ superpartial dislocations connected with an antiphase-boundary band [3]. The conditions for formation of Kear–Wilsdorf barriers are the high energy of antiphase boundaries in the primary octahedron (γ_0) and the low energy of antiphase boundaries in the cubic plane (γ_c), considerable anisotropy of elastic moduli, and a small separation of dissociated superpartial dislocations (SPD). The relative contribution of shear stresses in the cubic plane and the contribution to the driving force for the cross slip caused by separation of dissociated superpartial dislocations can be evaluated with the use of the parameters N and Q. The parameter N is the ratio between the Schmid factor in the cubic cross-slip plane and the Schmid factor in the primary octahedron, and Q is the ratio between the Schmid factor of the Shockley partials and the Schmid factor in the primary octahedron. These parameters qualitatively reflect the role of the cubic cross-slip plane and that of the separation of dissociated SPDs in the mobility of superdislocations in the primary octahedron and also the orientation dependence of the driving force for the cross slip of $a/2[\bar{1}01](111)$ screw superdislocations (see [3, 4]). Small separations of the screw SPDs are the condition for their cross slip and testify to variations in their mobility with varied shear stresses.

The relation between the deflection angle φ and the normalized crystallographic indices of the deformation axis in the stereographic triangle $[001]-[011]-[\bar{1}11]$ is schematically shown in Fig. 2. The angle φ characterizes the deflection of the deformation axis from the pole [001] toward the pole $[\bar{1}11]$ of the stereographic triangle. In the present article, the current value of the deformation direction φ coincides with the $[001], [\bar{1}39], [\bar{4}917], [\bar{2}34]$, or $[\bar{1}11]$ crystallographic directions. We assume that $[\bar{i}j1]$ are the normalized indices that define the direction of the deformation axis $[\bar{i}^* j^* k^*]$ in the stereographic triangle $[001]-[\bar{1}11]-[011]$. Then, the parameters N and Q depend on the indices $[\bar{i}j1]$ as

$$Q = \frac{-2i+j-1}{\sqrt{3}(-i+1)}, \qquad N = \frac{\sqrt{3}j}{i+j+1}.$$
(1)



Fig. 1. Yield stress (a) and thermal-hardening intensity versus temperature in Ni_3Ge single crystals deformed along the $[\bar{1}39]$ (b) and [001] and $[\bar{2}34]$ (c) crystallographic directions.

It follows from the diagram of Fig. 2 that the deformation-axis deflection angle φ is related to the normalized indices of the axis by the formula

$$\tan \varphi = \sqrt{i^2 + j^2}.\tag{2}$$

As is seen from formula (2), the deflection angle φ is a function of *i* and *j*. The indices *i* and *j* can be expressed in terms of *N* and *Q* from system (1). Apparently, the angle φ is also a function of *N* and *Q*.

Figure 3 shows the orientation dependence of the temperature increment $\Delta \tau = \tau (293 \text{ K}) - \tau (77 \text{ K})$ at the yield point on the parameter N in the octahedral plane (curve 1) and in the primary-cube plane (curve 2) (a) and the orientation dependence of the angle φ of deformation-axis deflection from the pole [0 0 1] toward the pole [$\bar{1} 1 1$] (b). The contribution caused by point defects was preliminary isolated from the dependence $\Delta \tau(T)$. In the orientation [$\bar{1} 3 9$], this contribution is rather substantial as compared to the orientations [$\bar{1} 1 1$], [$\bar{2} 3 4$], [$\bar{4} 9 17$], and [0 0 1]. The contribution was found by approximating the dependences $\tau_c(T)$ reported in [2]. On the basis of the dependence



Fig. 2. Determination of the orientation dependence of the deformation-axis direction [i j 1] in Ni₃Ge single crystals.



Fig. 3. Temperature anomaly of the yield stress in Ni₃Ge single crystals versus the orientation parameter N (a) and the deformation-axis deflection angle from the direction [0 0 1] toward the direction $[\bar{1} 1 1]$ (b).

of $\Delta \tau$ on the angle φ and on the parameter N, one can distinguish and analyze factors that govern the anomalous increase of the yield stress in the temperature range $\Delta T = 77$ –300 K. Such factors include thermally activated coupling of partial dislocations, enhanced contribution of shear stresses in the cubic plane with increasing φ , level of shear stresses in the octahedron, and the variation of the separation of dissociated superpartial dislocations under the action of shear stresses. The indicated qualitative conclusions can be drawn from variations of relative contributions of the terms with the parameters N and Q to the driving force of the thermally activated deceleration of screw superdislocations observed at a constant test temperature. As is seen from Fig. 3, the dependences $\Delta \tau(N)$ and $\Delta \tau(\varphi)$ display maximums of $\Delta \tau(T)$ near the orientation [$\bar{1}39$]. In crystals deformed along the [001] and [$\bar{1}39$] crystallographic directions at room temperature, if the slip occurs along the octahedral slip systems, these factors have a pronounced effect on mobility of dislocations. In the [$\bar{4}917$], [$\bar{2}34$], and [$\bar{1}11$] orientations, the peak temperature is $T_p \approx 300$ K; as a result, the slip along the cubic systems turns out to be preferable. The influence of orientation on manifestation of thermally activated locking of octahedral superdislocations in the orientations [$\bar{4}917$], [$\bar{2}34$], and [$\bar{1}11$] turns out to be minimal at room temperature.

An additional factor that indicates an important role of cubic slip systems in mobility of superdislocations in the octahedron planes is the orientation dependence of the peak temperature (T_p) in the positive dependence of the yield stress. Calculations of the orientation dependence of T_p based on the assumption of identical shear



Fig. 4. Orientation dependence of the peak temperature T_p of the anomaly and that of the yield stress $\tau_c(T_p)$ in the octahedron plane: the filled and open points refer to experimental and theoretically predicted values, respectively.



Fig. 5. Temperature dependences of the fraction of rectilinear dislocations in differently oriented Ni_3Ge single crystals under 5-% deformation.

stresses in the cube and in the octahedron were reported in [5]. It follows from these calculations that the peak temperature decreases as the angle of $[\bar{1} 1 1]$ is approached. Figure 4 shows the temperatures T_p in Ni₃Ge single crystals. Indeed, as the orientation of the deformation axis approaches the direction $[\bar{1} 1 1]$, the Schmid factor of the cubic slip system increases and the peak temperature of the anomaly decreases. It should be noted, however, that the peak temperature T_p for the orientations [001] and $[\bar{1}39]$ indicated in Fig. 4 is determined by locking of superdislocations not only via the Kear–Wilsdorf mechanism but also by condensation of point defects on the edge components.

An analysis of the dislocation substructure in the examined Ni₃Ge single crystals showed that, up to their mechanical failure, the dislocation structure remains random and uniform. The main configurations in the dislocation structure are rectilinear dislocations and bent superdislocations, dipoles and dipole configurations, and fragments. Dipoles insignificantly contribute to the flow stress. The density of rectilinear dislocations increases with increasing strain and test temperature in the temperature range T = 77-293 K. Moreover, the relative fraction of these dislocations increases with increasing temperature in the low-temperature interval of the anomaly (Fig. 5). The fraction of rectilinear dislocations in the orientations [001] and [$\overline{1}39$] in Ni₃Ge single crystals deformed to $\varepsilon = 5$ % was defined as the ratio of density of rectilinear dislocations to scalar density. A comparison of the relative fraction of rectilinear dislocations (Fig. 5) with the temperature dependences of the yield stress $\tau_c(T)$ (see Fig. 1a) reveals a correlation between the accumulation of density of such dislocations and the low-temperature anomaly of



Fig. 6. Diagrams illustrating the formation (a) and breakdown (b) of Kear–Wilsdorf barriers on the screw components of superdislocations in Ni_3Ge single crystals.

the yield stress. The increase in the fraction of rectilinear dislocations indicates their substantial contribution to the thermally activated growth of yield stress. The positive temperature dependence of the flow stress in this range of temperatures can be attributed to the increase in density of rectilinear dislocations. There are many reported data showing that rectilinear dislocations are Kear–Wilsdorf barriers [1, 3, 4, 6–10]. Low mobility of Marcinkowsky superdislocations leads to elevated density of rectilinear dislocations and to a high fraction of Kear–Wilsdorf barriers associated with rectilinear dislocations in Ni_3Ge single crystals.

Estimation of the Driving Force of Formation and Breakdown of Kear–Wilsdorf Barriers. Various configurations of the Kear–Wilsdorf barriers were analyzed in detail in [3, 8, 9, 11–13]. It was shown that anisotropy of elastic moduli in the alloys examined, the energy of antiphase boundaries (APB) in the octahedron and in the cube, and the mutual relation between them play an important role in formation of barriers on Marcinkowsky superdislocations. It should be noted, however, that the balance between the driving forces of the hypothetical configurations of the Kear–Wilsdorf barriers on the leading or led undissociated superpartial dislocations considered in the models of [3, 8, 11, 12] cannot be correlated with the experimentally found driving force for the thermally activated growth of the yield stress. The primary reason is that the driving forces were calculated considering the barrier configurations at the final stage. Yet, the thermally activated part of the formation process for Kear–Wilsdorf barriers is restricted to coupling of the Shockley partials at the moment the barrier passes into the cubic cross-slip plane. The reported configurations of screw superdislocations on which barriers form only testify to strength and stability of Kear–Wilsdorf barriers, which substantially contribute to strain hardening of materials.

To analyze the thermally activated coupling of Shockley partials on the leading superpartial dislocation, we use the calculation scheme of [8, 9]. We assume that the superdislocation resides in the primary octahedron (Fig. 6a). This difference from the Hirsch schemes [8, 9] is substantial because it implies thermofluctuational coupling of partial dislocations. The leading superpartial dislocation at a given time undergoes fluctuation-induced coupling and becomes capable of moving in the $(0\,1\,0)$ direction; i.e., this dislocation occupies the position on the top of the potential barrier. This passage into the cubic plane is affected by anisotropy of elastic moduli and by shear stresses in the cube. The driving force on the leading undissociated superpartial dislocation (the positive direction is indicated in Fig. 6a) is

$$F_d = F_r \cos \alpha + F_\theta \sin \alpha - \gamma_0 + \tau_c^{(010)} b + \tau_F^{(010)} b + K\tau.$$
(3)

Here, F_r and F_{θ} are the radial and tangential components of the force of interaction between superpartial dislocations, α is the angle between the octahedron and cube planes, γ_0 is the antiphase-boundary energy in the octahedron, $\tau_c^{(010)}$ is the shear stress in the cube, and $\tau_F^{(010)}$ is the self-locking stress of the superpartial dislocation. The first two terms determine the projection of the interaction force onto the positive direction (see Fig. 6a). For the screw components of undissociated superpartial dislocations [12, 13], the tangential and radial components are related by the formula $F_{\theta} = F_r(A \sin 2\alpha/(2((A-1)\cos^2\alpha + 1))))$, where the parameter A reflects the anisotropy of elastic moduli. The last term in (3) takes into account the Escaig effect [3, 9, 11], which determines the tensioncompression asymmetry, and the orientation dependence of the yield-stress anomaly. An analytical expression for K was derived in [11]. Under dynamic equilibrium on undissociated superpartial dislocations, we have [11]

$$F_r = \gamma_0 + \tau_c^{(111)}b.$$
(4)

We substitute F_r into (3) and obtain after simple transformation

$$F_d = (\gamma_0 + \tau_c^{(111)}b)(A\cos\alpha/((A-1)\cos^2\alpha + 1)) - \gamma_0 + \tau_c^{(010)}b + \tau_F^{(010)}b + K\tau.$$
(5)

Formula (5) yields the driving force for the temperature growth of the yield and flow stresses. Equation (5) has singularities. This equation does not contain the energy of the antiphase boundary in the cubic cross-slip plane and the term caused by anisotropy of antiphase boundaries (i.e., $\gamma_0 - \gamma_c$) in the primary octahedral and cubic cross-slip planes. Hence, there are no realized criteria for the temperature yield-stress anomaly in materials with $L1_2$ related to anisotropy of antiphase boundary energy in the octahedron and in the cube. Such criteria were suggested in [3, 11, 12]. At the moment under consideration, the leading undissociated superpartial dislocation is still outside the cubic cross-slip plane.

The driving force for the formation of Kear–Wilsdorf barriers was found for the following parameters of Ni₃Ge single crystals deformed along the [$\bar{1}39$] crystallographic direction: $\gamma_0 = 180 \text{ mJ/m}^2$, A = 1.61 [10], b = 0.25 nm, $\tau_c^{(111)} = 567 \text{ MPa}$, and $\tau_c^{(010)} = 230 \text{ MPa}$. The dislocation self-deceleration stress in the cubic plane $\tau_F^{(010)}$ was determined experimentally, based on the linear dependence of the shear stress $\tau = \tau_F + \alpha G b \rho^{0.5}$ on the dislocation density $\rho^{0.5} \tau_F^{(010)} = 167 \text{ MPa}$. The estimates yield the value of the driving force $F_d \approx 0.03 \text{ eV/atom}$, which is close to the experimental values reported in [5]. It should be noted that taking into account non-locality of Kear–Wilsdorf barriers [3, 4, 9] and breakdown of barriers by superkink motion [3, 9] adds an expression of the type exp $(-F_d/(3kT))$ in the Arrhenius relation. In the model of [6], the driving force is also decreased by a factor of three. Considering the indicated numerical value, the driving force can be considered as a good approximation to the experimentally found value of the driving force for the temperature growth of the yield stress in Ni₃Ge in the [$\bar{1}39$] orientation of the low-temperature anomaly, equal to 0.010 eV/atom [2].

It seems possible to use formula (5) in the analysis of the orientation dependence of the yield and flow stresses in the simplest case. In this analysis, we restrict ourselves to finding the extremum with respect to N. Here, the formation of the Kear–Wilsdorf barrier is assumed to occur at a point. This means that finding the extremum of the function $\Delta \tau(N)$ reduces to finding the extremum of $F_d(N)$. Let us find the dependence of the Schmid factors on the parameters N and Q for the cube $\chi^{(010)}$ and for the octahedron $\chi^{(111)}$. Passing to normalized indices in the Schmid factors and then determining these indices from formula (1), we find the desired dependence. After insertion of the found functions $\chi^{(010)}(N,Q)$ and $\chi^{(111)}(N,Q)$ into formula (5), under the assumption that $\partial F_d/\partial N = 0$, this equation yields the following values of these parameters: $N \approx 0.25$ and $Q \approx 0.7$. It follows from system (1) that N = 0.3 in the [$\bar{1}39$] orientation. The found extremum value is close to the value of N in the [$\bar{1}39$] orientation. This means that the extremum in orientation dependences of thermal hardening of Ni₃Ge single crystals lies near the [$\bar{1}39$] orientation.

Let us consider now the driving force for the breakdown of the Kear–Wilsdorf barrier due to activated slip in the cubic cross-slip plane. The calculations are also based on the Hirsch model [7, 9]. Figure 6b shows a schematic of the barrier (with the positive direction indicated). The driving force on the leading superpartial dislocation is

$$F_d = -\gamma_c + F_r \cos\left(\alpha - \beta\right) + F_\theta \sin\left(\alpha - \beta\right) - \gamma_{\rm CSF} + \tau_{\rm PS}b + \tau_c^{(010)}b - \tau_F^{(010)}b. \tag{6}$$

The first and fourth terms here are the energy of the antiphase boundary in the cube and the energy of the complex stacking fault in the octahedron, the second and third terms describe the interaction of the leading and led superpartial dislocations, and the remaining terms describe the contribution due to shear stresses in the cube $(\tau_c^{(010)})$, the contribution of friction-induced superdislocation stress $(\tau_F^{(010)})$, and the interaction of the Shockley partials of the leading superpartial dislocation (τ_{PS}) . As in estimating the driving force for Kear–Wilsdorf barrier formation, the calculations by formula (6) imply that, owing to thermal activation, the leading superpartial dislocation resides on the top of the potential barrier. Apparently, at the cross-slip moment, the angles α and β coincide. The calculations of the driving force by formula (6) are approximate calculations because the led undissociated dislocation may reside either in the cube or in the octahedron. In the latter case, cross-slip of the led superpartial dislocation readily occurs at the temperature T_p at which the breakdown of the Kear–Wilsdorf barrier is observed. The led undissociated superpartial dislocation is not at dynamic equilibrium, and formula (4) cannot be used to calculate F_r in the cube;

hence, $F_r \approx Gb^2/(2\pi r)$. Owing to anisotropy of elastic moduli, the led undissociated superpartial dislocation returns into the cubic cross-slip plane under the action of $F_{\theta} \neq 0$. The separation of dissociated superdislocations in the cube is $r \approx 6.5$ nm, and $G = 8 \cdot 10^{10} \text{ N/m}^2$. We assume that $\gamma_{AFB} \approx 180 \text{ mJ/m}^2$ in the octahedron, $\gamma_{AFB} \approx 140 \text{ mJ/m}^2$ in the cube, $\tau_c^{(111)} \approx 560 \text{ MPa}$, and $\tau_c^{(010)} \approx 265 \text{ MPa}$. We insert these values into (6) and obtain $F_d \approx -0.145 \text{ eV/atom}$. It should be noted that the driving force F_d for the indicated parameters is negative; hence, the driving force describes the thermally activated softening of the flow stress. The predicted value of the driving force for Kear–Wilsdorf barrier breakdown is close to the experimental value of the driving force for temperature softening of the yield stress above the peak temperature of the anomaly. The experimental value of the driving force was found by fitting the curves of $\tau_c(T)$ in [139]-oriented Ni₃Ge single crystals [2] with an Arrhenius-type relation.

Summary. Thus, the present study of the temperature dependence of the yield stress has revealed a complicated nonmonotonic behavior of the flow and yield stresses with temperature. The low-temperature yield-stress anomaly is attributed to the formation of Kear–Wilsdorf barriers on mobile superdislocations. This conclusion is supported by electron-microscopy observations of dislocation substructures, by the data on the low-temperature anomaly, and by the dependence of this anomaly on the orientation of the deformation axis in Ni₃Ge single crystals. Calculations of the driving force for formation and breakdown of Kear–Wilsdorf barriers, performed with allowance for thermofluctuational coupling of partial dislocations show the model proposed to agree well with the experimental values of the driving force. In the present model, the orientation dependence of the yield-stress anomaly with an extremum near the orientation $[\bar{1}39]$ is naturally taken into account.

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