Crystal interface and high-resolution electron microscopy—the best partner

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Abstract

Several contributions of HRTEM on the interface science are reviewed in chronological order. The first contribution of HRTEM is the observation of gold (113)S11 boundary, giving experimental proof of the CSL model. An observation of the asymmetric (112)S3 boundary follows. A SiC grain boundary is effectively assessed not by the density of CSL point but the number of dangling bonds in the boundary. A ZnO/Pd interface provides an example that a misfit dislocation does not necessarily accommodate the lattice mismatch. Segregated interface shows characteristic HRTEM image contrast, suggesting change in atomic bonding. An atomic height step in the semiconductor hetero interface is observed by the Chemical Lattice Image technique. In the diamond grain boundary a dangling bond may not elevate the boundary energy, being contradictory of the least dangling bond rule. Super-high resolution of the HVHRTEM enable us to determine atomic species in the grain boundary. Combined use of HRTEM and EELSE allows us to discuss the correlation between atomic structure and nature of the corresponding interface. It is not exaggeration to say that modern interface science does not exist without HRTEM. On the other hand, many complicated interfaces found by HRTEM remained as unanswered questions. An innovative structural model is requested to appear on the scene. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Crystal interfaces had been locked in a kind of “black box” until the first decade of the latter half of this century. Lack of an experimental tool for atomic structure observation caused such a poor situation. The innovative invention of the X-ray diffraction method did not profit the crystal interface in spite of its brilliant success in crystal structure analysis. The unfruitful result of the X-ray diffraction was attributed to the weak interaction of X-ray with an atomic potential. The crystal interface was too small to diffract enough X-ray waves for detection. Accordingly, an available means for the interface structure analysis, during that period, was narrowed down to the inductive reasoning from the response of a polycrystalline material to the external mechanical stimulus. The “behavior”, in a sense, was the only clue to analyze the “structure”.

Despite such experimental inconveniences of that time many researchers proposed a wide variety of structure models and theories [1–3]. However, most of them do not currently survive except for two geometrical models—the o-lattice model proposed by Bollmann [4–7] and the coincidence-site-lattice (CSL) model of Brandon [8]. It should be noted that the survivors were constructed not by the inductive reasoning from such an indirect experimental information but purely from a mathematical consideration. Poor experimental outcomes forced the use of theoretical considerations.

A mighty tool that opened the “black box” was the electron microscope. Employing an electron microscope, Ishida [9] observed a grain boundary dislocation which was predicted by the CSL boundary model of Brandon [8]. Gleiter [10] and Levy [11] also observed the grain boundary dislocation in a metallic CSL boundary. These observations, though rather indirect, supplied the first experimental support for the CSL model. The tool that finally opened the door to a new era of the interface (or grain boundary) science was the high-resolution transmission electron microscope (HRTEM), which produced lattice images. By this method one can directly observe the periodic atomic structure of crystal interfaces even if an interface consists...
of only a few atomic layers. The strong scattering nature of electron from the atomic potential, corresponding to $10^4$ times that of X-ray, and the strong interaction with the electro-magnetic field which enables to form a "microscope of electron" led us to the breakthrough.

A recent high voltage HRTEM established at The University of Tokyo was capable of obtaining the ultimate sub-angstrom resolution [12]. The super high resolution of the high voltage high-resolution electron microscope (HVHRTEM) enables us to distinguish even the atomic species in the grain boundary by image contrast (Fig. 1). Modern HRTEM is going to further open up the new era of interface.

In the present paper, the several contributions of high-resolution electron microscopy to the interface structure analysis are shown in chronological order.

2. First observation of $\Sigma 11$ CSL boundary

Observations of the grain boundary dislocation by the electron microscope partly provided experimental evidence for the CSL model. The CSL model allows a small deviation from the exact coincidence orientation to be complemented
by a secondary dislocation (grain boundary dislocation (GBD)) lying in the boundary at a certain interval.

After these observations, the major concern of interface researchers was to find the final direct proof of the CSL boundary model, i.e. by direct observation of the atomic structure of the CSL boundary. This was possible with the HRTEM. Fig. 2 shows the first picture of the (113)\textsubscript{S}11 CSL grain boundary which is one of most stable CSL boundary. The picture was taken employing an HVHRTEM of The University of Tokyo [13]. Each bright spot in the picture represents an atomic site. Every condition predicted by the model is shown in the picture; bright spots are geometrically arranged showing a good correlation with the CSL model (CSL points lying along the boundary). The boundary plane is parallel to the (113) plane which is the densest CSL plane of the corresponding $\Sigma$ value.

Critical assessments of the CSL model obtained using HRTEM were as follows: (1) validity of the model partly fails at high $\Sigma$ values; and (2) if the boundary plane is parallel to the secondary dense CSL plane an atomic structure of the CSL boundary becomes rather unstable even at small $\Sigma$ values (Fig. 3) [14]. If an atomic distance at the interface is much closer than that of the nearest neighbors in the crystal, the integrated repulsion force of the atomic pairs facing the boundary makes the crystal displace along the interface leading to an asymmetric atomic structure. A major cause for this partial failure is that this geometrical model has no physical content.

3. Least dangling bond rule of ceramic grain boundary

In ceramics, the CSL model cannot be a major guiding rule for the assessment of the grain boundary atomic structure. Complicated elemental composition, large size of the unit cell and non-homogeneous directional atomic bonding would prevent the boundary structure from matching well with the simple geometrical model.

Fig. 4 [15] shows one of the lowest energy grain boundaries observed in an $\alpha$-SiC bi-crystal that was grown by the sublimation–re-crystallization method. Since the bi-crystal grows very slowly in the free space, without any external applied force, the grain boundary holds the most stable atomic structure through the growth period. The morphology of the bi-crystal is a “T” shape; Two platelet crystals, 4 mm in edge length and 0.3 mm in thickness, are joined by the (0001) plane of one crystal and the (112\text{\textbar}0) plane of the other. This boundary is not symmetric any more.

Each bright spot in the picture corresponds to an atomic
tunnel at the present imaging conditions. Atoms lie in the dark contrast. A periodic boundary unit consists of six bright spots, which are a large spot, a small spot and four ordinary size spots. The most reasonable (in physics) atomic structure model is superimposed in Fig. 5 [15] showing the best matching. A seven-member atomic ring, a five-member ring and a six-member ring, respectively, correspond to, a large, a small and an ordinary sized bright spots in the picture. Two three-coordinated atoms are necessary to complete the boundary’s atomic structure, but it is shown that this number is the smallest amongst the likely candidates of the structures. In the covalent bonding material, the number of three-coordinated atom and the structural stability shows a tight correlation [16]. This tight correlation is attributed to the higher energy level of a dangling bond (sp₀ orbital electron) of the three-coordinated atom rather than to the sp³ electron of the four-coordinated atom in the crystal. A more dangling bond may cause higher grain boundary energy. Good correlation of the HRTEM picture with the structural model that contains the least dangling bond shows that this boundary holds the lowest atomic structure energy, as can be expected from the growing process.

4. Atomic structure of the metal/ceramic interface

4.1. ZnO/Pd interface [17]

The metal/ceramic interface is discussed in a more general way. The lattice mismatch produced at the interface is a major subject in structure assessment. The popularly employed model for the discussion of this subject is the o-lattice model (or o-lattice theory) proposed by Bollmann [4–7]. The o-lattice model suggests that the lattice mismatch produced at the interface should be complemented by introducing a secondary dislocation called “misfit dislocation”. The idea of misfit dislocation is purely based on geometry with no physical content. Although we can define a misfit dislocation in any interface, it is not apparent if the misfit dislocation really accommodates the elastic stress in the interface. The o-lattice model may face the same limitation as the CSL model in the case of real materials.

In real materials, a misfit dislocation is actually observed in many cases; the example of a zinc-oxide/palladium (ZnO/Pd) system is shown in (Fig. 6) [18]. Here, the slant interface is indicated by means of an arrow. An extra half-plane of the misfit dislocation is observed at every nine Pd planes, on the average, along the interface. A gradual atomic displacement corresponding to the strain field of the dislocation is observed along the extra half plane. A wavy atomic displacement is observed along the interface too, making this a three (two)-dimensional strain field of dislocation. This interface is parallel to the \(\{1102\}\) plane of ZnO and the \(\{002\}\) of Pd. The \([1120]\) axis of ZnO and the \([110]\) of Pd are parallel to the electron beam.

On the other hand, the misfit dislocation is frequently invisible in an HRTEM picture, even though the joined crystals are apparently different in both the lattice parameter and the crystal structure. Fig. 7 shows an interface of ZnO...
Both the [112¯0] axis of ZnO and the [110] of Pd are also parallel to the electron beam. The atomic structure of this system is determined by employing both, the molecular dynamic calculation and the image simulation. The (0001) plane of ZnO is terminated not by O but by Zn at the interface. The interface shown by two arrows is parallel to the (0001)ZnO and the (111)Pd. On the ZnO side, each of the white spots do not change their original site, i.e. no atomic displacement is recognized in the picture. In the Pd side also all the spots, except for that of the (111) plane next to the edge of ZnO crystal, were not displaced. Atoms belonging to the mono-atomic row next to the ZnO crystal irregularly change their site in the plane parallel to the interface or the (111) plane. As a result, no atomic displacement corresponding to a misfit dislocation is observed in the micrograph.

Under this situation, the atomic distance of some atoms facing the interface apparently exceeds the equilibrium distance. If the temperature is elevated, further expansion of the atomic distance must take place due to the difference in thermal expansion coefficient between the two crystals, and it leads to the breakdown of atomic bonding at the interface. If there were misfit dislocations present, the thermal stress is easily relieved by their slide motion. The weakness of the metal/ceramic interface against cyclic thermal stress is attributable to the absence of a relaxation mechanism in the misfit dislocation.

An important problem is that the mismatch accommodation takes place in the (1102)ZnO/(002)Pd interface, but it does not do so in the (0001)ZnO/(111)Pd interface. Apparently, the difference between these contradictory interfaces is only due to the interface orientation. The orientation dependence of both the bonding strength across the interface and the rigidity of the joined component crystal could be major controlling factors for the lattice mismatch accommodation.

4.2. Hydrogen segregated MgO/Pd interface [19]

The bonding state reconstruction related to the atomic structure change due to hydrogen segregation in the magnesium-oxide/palladium (MgO/Pd) interface was observed by HRTEM.

In the ordinary HRTEM analysis, an HRTEM picture obtained is compared with a simulated image that was calculated from the previously constructed probable model.
structure, and then the difference is extracted. The extracted difference between the picture and the simulated image is fed back to improve the model structure. The simulated image of the improved model is compared with the picture again. This procedure is repeated until the best matching between the picture and the simulated image is attained. The minimum requirement for the success of this method is that a starting model of the object structure is either known or deducible. For the segregated interface this is typically not the case. No experimental information was available to build up a starting model of the hydrogen segregated MgO/Pd interface for the image calculation. Therefore, the first thing to do for the structure analysis of the hydrogen segregation by the HRTEM was to confirm the presence of hydrogen in the interface.

Segregation in the interface is confirmed in the following way. Hydrogen is first injected by electrolytic method and then the specimen is heated to evacuate the hydrogen. When heated at 800°C for 30 h all the hydrogen in the crystal and those caught by the strain field of lattice defects are evacuated. It is supposed that the remainder is either chemically trapped or are segregated in the interface. The evacuated amount is then subtracted from the amount of injected hydrogen and the remainder divided by the area of the interface. The final amount of segregation corresponded to the amount in one mono-layer of the interface.

Each of the pictures in Fig. 8 shows an original and a segregated interface. The image contrast of the interface region differs between the two pictures. The segregated interface (Fig. 8b) looks brighter and rather diffused in comparison to the original interface (Fig. 8a.). Since the imaging condition is adjusted to show an atomic site (low potential site) this looks bright. The brighter contrast of the segregated interface suggests that the local density of the electron in the interface has increased. The segregated hydrogen may attract electrons of the neighboring atoms. Another change of the segregated interface is an expansion of the plane distance of the interface by 20%. The classic rigid ball model, which filled the free volume of the interface with a ball, can hardly explain the phenomenon. The hydrogen ball is too small to take into account the interface expansion by 20%.

In order to deduce a possible cause of the bright contrast and the 20% expansion of the segregated interface an image simulation was performed. In Fig. 9, the original picture, simulated images and figures of their equal brightness contour are shown. (1), (2), (3) and (4) in the figure corresponds, respectively, to an original micrograph of the

Table 1
(All the diffractions, except for 200 and 024, are given by the square of the sum of each atomic scattering factor or by the sum of the squared atomic scattering factors)

| (hkl) | I_{GaAs} | I_{AlAs} |
|------|----------|----------|
| 100  | 0        | 0        |
| 200  | 16(f_{Ga}^2 - f_{As}^2) = 0 | 16(f_{Al}^2 - f_{As}^2) = 0 |
| 400  | 16(f_{Ga}^2 + f_{As}^2) | 16(f_{Al}^2 + f_{As}^2) |
| 011  | 0        | 0        |
| 111  | 16(f_{Ga}^2 + f_{As}^2) | 16(f_{Al}^2 + f_{As}^2) |
| 211  | 0        | 0        |
| 311  | 16(f_{Ga}^2 + f_{As}^2) | 16(f_{Al}^2 + f_{As}^2) |
| 022  | 16(f_{Ga}^2 + f_{As}^2) | 16(f_{Al}^2 + f_{As}^2) |
| 024  | 16(f_{Ga}^2 - f_{As}^2) | 16(f_{Al}^2 - f_{As}^2) |

Fig. 11. Diffraction intensity change due to thickness change in GaAs and AlAs crystals. The 220 diffraction of GaAs is almost zero through the thickness. The others show a sinusoidal change.

Fig. 12. An image simulation of AlAs and GaAs viewed from (100) axis. Each bright spot corresponds to the respective atomic site in AlAs but it appears alternately in GaAs.
5. An atomic step of the GaAs/AlAs hetero-interface

The GaAs/AlAs hetero-interface is one of the most challenging materials for high-resolution electron microscopy. The requested task was to assess the interface roughness in the atomic dimension. The trouble is that no distinctive feature is detectable in the HRTEM image of the materials viewed from the popular \( k \overline{1}0 \) axis. Since the HRTEM image contrast is given by the phase shift of elastic diffraction waves which does not contain any chemical information, those materials that are same in their lattice parameters cannot be distinguished from each other in the image contrast, even if they differ in their chemical composition. The structural information provided by HRTEM is nothing but the geometry of lattice. Actually, the HRTEM image of the GaAs/AlAs hetero-junction observed in the direction parallel to the \( \langle 110 \rangle \) axis looks like a single crystal (Fig. 10). The GaAs/AlAs hetero-interface is undetectable even by the HRTEM, in principle, when viewed from the \( (110) \) axis [20].

The difficulty was to overcome by careful analysis the diffraction intensity in the materials. The diffraction amplitude in the crystal is shown by crystal structure factor \( F_{(hkl)} \), which is given by the equation

\[
F_{(hkl)} = \sum_j f_j(\theta) \exp\left\{ -2\pi i(hu_j + kv_j + lw_j) \right\}
\]

where \( f_j(\theta) \) is the atomic scattering factor. The calculated diffraction intensity \( I = |F_{(hkl)}|^2 \) is shown in Table 1 for low indices \( (hkl) \) [20]. All the diffractions, except for 200 and 024, are given by the square of the sum of each atomic scattering factor or by the sum of the squared atomic scattering factors. The available diffractions for HRTEM imaging are ordinarily limited to 111, 200 and 220 due to the resolution power of the HRTEM which is at most 0.14 nm. In order to be useful, the 200 diffraction has to be proportional to the difference in the component atomic scattering factors.

Employing the popular \( (110) \) illumination, an image contrast is mainly contributed by the 111 diffraction waves that show the highest intensity in the fcc system. The 111 diffraction intensity, which is proportional to the sum of each atomic scattering factor, is not affected by the difference in the atomic scattering factors. Unfortunately, the efficiency of the 200 and the 220 diffraction waves is very small compared to that of the 111. Therefore GaAs and AlAs look totally similar in the HRTEM image.

When illuminated from the \( (100) \) direction, the 200 diffractions contribute mainly to the image contrast. Fig. 11 shows an intensity change of diffraction waves due to the specimen thickness change in GaAs and AlAs. In GaAs, the 200 diffraction intensity, which is proportional to the difference of each of the atomic scattering factor, is almost zero at all thickness in the GaAs. The extremely weak intensity of the 200 diffraction is attributed to the extremely close atomic scattering factors of Ga and As. On the other hand, in

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Fig. 13. A chemical lattice image [20] of an artificial GaAs/AlAs hetero-junction. The interface is recognized by the transience of image pattern from one to the other, and is shown by an arrow.

Fig. 14. Mono-molecular step in height in the artificial GaAs/AlAs interface present between P1 and P2. Tracing the interface from the left, the white spots became diffuse at P1 and disappeared in the line at around P2 where the interface changed to the next line.
AlAs the 200 diffraction wave maintains finite intensity showing a sinusoidal change. Other diffraction waves that may contribute to the image formation show a sinusoidal intensity change due to the specimen thickness change and have finite intensity in the thickness region from 5 to 15 nm. If the specimen thickness, therefore, is controlled between 5 and 15 nm each diffraction wave, except for 200 of GaAs, would have finite intensity. Under this condition the HRTEM image of GaAs and AlAs possibly differ from each other.

Following the above principle, an image viewed from $k_{100}$ zone axis was simulated (Fig. 12). In the figure, each bright spot of the AlAs corresponds to an atomic site, but in the GaAs a bright spot appears alternately. Now we can distinguish GaAs and AlAs by the image contrast pattern originated by the differences in the chemical composition. Fig. 13 shows a picture of a GaAs/AlAs hetero-junction taken by this imaging technique. An interface is actually recognized by the transience from one image pattern to the other and is indicated by arrows.

This newly developed technique was applied to analyze the interface roughness in the atomic dimension (Fig. 14). An interface in the picture does not lie on the same level showing that an atomic step present. Starting from the arrow of the left-hand side we can trace clear bright spots at constant spacing, but in the middle the bright spot becomes diffused (P1) and then disappears (P2). In order to trace the bright spot at the same spacing we must move to the next row. Here there is an atomic step that is oblique to the surface in the region where the bright spots are diffuse. A schematic illustration is shown in Fig. 15. The observed step is deduced to be parallel to the (110) plane.

6. Diamond grain boundary

6.1. Failure of the “least dangling bond rule”

It was mentioned in Section 3 that any grain boundary in the covalent bonding material, such as silicon, germanium and silicon carbide (SiC), is supposed to contain the least amount of dangling bonds (three coordinated atom) that cause the elevation of the grain boundary energy. A $\Sigma 9$ boundary, for example, is completed without any surplus and shortage of bonds, or without any dangling bond, in the boundary being parallel to the (221) plane. But if the boundary plane is parallel to the (114) plane at the same crystal orientation, four dangling bonds must be introduced in a unit length of the boundary (Fig. 16) [21] so that the (114) $\Sigma 9$ CSL boundary is considered to be unstable. Actually, the $\Sigma 9$ boundary tends to be parallel to the (221) plane in silicon and germanium [22].

The diamond grain boundary, however, is rather contradictory to this empirical guiding rule of the covalent bonding material’s boundary. Most of the $\Sigma 9$ boundaries in diamond are parallel not to the (221) plane but to the (114) plane (Fig. 17) [23]. The parallelism of the $\Sigma 9$ boundary to the (114) plane tells that the “least dangling bond rule” is not valid for every covalent bonding materials.

6.2. Electron structure of the diamond grain boundary

Inconsistency of the diamond grain boundary with the other covalent bonding material’s grain boundary was
clearly explained by employing electron energy loss spectroscopy (EELS). An EELS spectrum of a diamond grain boundary is shown in Fig. 18 [23], in which three spectra are shown together. The uppermost spectrum is acquired from a region containing a grain boundary. The middle one is from the neighboring crystalline region. The major line at 291 eV corresponds to the $sp^3$ state of diamond (or anti bonding state of $sp^3$ orbital electron). Both the spectra were normalized by the intensity of the major $sp^3$ line and then subtracted from each other. The remainder spectrum is put at the bottom, showing the net EELS signal from the grain boundary. The grain boundary spectrum is characterized by a line located at 285 eV which corresponds to the $\pi^*$ orbit of carbon, the conjugate of $\pi$ orbit. This $\pi^*$ line of carbon appears whenever any three-coordinated atom is contained in the boundary. The boundaries that show the EELS $\pi^*$ line are very popular in diamond [24]. Simultaneity of the EELS $\pi^*$ line and the three-coordinated atom of the diamond grain boundary suggests that a “dangling bond” ($sp_z$ state) of the three-coordinated atom easily transforms into the $\pi$ state, without remaining as a dangling bond different from the other covalent bonding materials. The lower energy level of the $\pi$ orbit in carbon must be a major cause. The reconstructed $\pi$ electron does not elevate the boundary energy any further. This is the reason why an empirical “least dangling bond rule” is not valid in the diamond grain boundary.

It should be noted that the grain boundary structure need not be the same even if both the crystal structure and the bonding manner of the crystal are common. The failure of
the “least dangling bond rule” means that the grain boundary structure is finally governed by the characteristics of electron structure of each atomic species (element).

7. Super high-resolution analysis of a grain boundary [25]

In order for binary element material to determine a stable boundary structure the chemical structure must be known, since the site exchange of each element in the grain boundary may cause the change in the boundary energy even in the same geometry. The recent development of a super high-resolution transmission electron microscope (SHRTEM) enables us to observe the real atomic structure and to distinguish the chemical element in image contrast as shown in Fig. 1, where silicon and carbon is imaged at different contrasts [12].

The bottom curve in Fig. 19(a) shows a darkness profile of the Si–C dumbbell in the SHRTEM picture shown above. Two peaks in the curve corresponding to silicon and carbon are apparently recognized. The simulated image in Fig. 19(b) also shows an almost similar profile. The atomic distance of the dumbbell, distance from peak to peak, is 0.01 nm. The figure shows that the atomic site of each element is quantitatively distinctive in the HRTEM image. The first determination of real atomic structure of the SiC grain boundary by SHRTEM is shown in Fig. 20. The atomic site of each element is exactly determined in the figure. From the analysis two points are concluded: one is that the like pair in the boundary is the Si–Si pair; and the other is that both the three-coordinated atoms located at the five- and seven-member rings are carbon. The latter term is especially consistent with the result of the EELS measurement of the diamond grain boundary. The energy reduction mechanism due to the electron orbital transition from sp to \( \pi \) of carbon seems to be working in the SiC boundary too.

8. Summary

Questions asked in the last hundred years were almost answered by high-resolution transmission electron microscope (HRTEM) in the last 20 years. Most of the grain boundaries of fcc metals, bcc metals, diamond structure materials, ceramics and others were observed by HRTEM. Nowadays, the correlation between atomic structure and the nature of the corresponding interface is discussed by the combined use of HRTEM and electron energy loss spectroscopy. A new area of study, grain boundary engineering, based on the previously accumulated knowledge may come into existence. It is not an exaggeration to say that modern interface science does not exist without HRTEM. On the other hand, many complicated interface structures that were found by HRTEM remain unexplained. The two geometrical models, coincidence-site-lattice, \( \omega \)-lattice models, whose limitations are pointed out here using HRTEM, also remain without any improvement or alternative suggestions. Many advanced materials, complicated in both crystallography and chemistry, are waiting for a new model to be proposed for the interface analysis. At present, an innovative interface structure model is yet appear on the scene.

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