# Excess Volume of the Solid/Liquid Interface in Fe-6 at.%Si Bicrystals Wetted by Liquid Zinc

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Abstract. The pressure effect on grain boundary wetting in Fe-6 at.%Si bicrystals of different misorientation angles but constant misorientation axis has been studied. The wetting agent was liquid zinc. It was found that the pressure for the dewetting transition is higher for the near  $\Sigma$ 5 boundary than for the other general boundaries, where  $\Sigma$  is the inverse density of the coincidence sites in the two misoriented crystal lattices. This result was explained assuming a thinner liquid film wetting the near  $\Sigma$ 5 boundary than in the case of nonperiodic grain boundaries. Furthermore, the wetting angle increased with increasing pressure. The wetting angle dependence on pressure could be understood assuming a excess surface volume of the solid/liquid (S/L) interface higher than 0.2 nm. This is considerably higher than the estimated excess volumes of grain boundaries based on computer simulations. To explain this result, it was postulated that in the system studied, where diffusion of Zn, Fe and Si perpendicular to the S/L interface takes place, the S/L interface is relatively thick and the interaction between the two crystals separated by the melt extends over more than 2 nm distance. This long-range interaction was rationalized in terms of clusters of several atoms, detaching from the solid and dissolving in the melt at some distance from the bulk.

Keywords: grain boundary energy, diffusion, segregation, solid/liquid interface, wetting

#### 1. Introduction

Grain boundary (GB) wetting takes place at the intersection of GBs and the solid/liquid (S/L) interface [1-28]. In some cases perfect wetting takes place and a thin liquid film penetrates along the GBs. This phenomenon is called the wetting transition and may considerably accelerate the reaction between the elements in the liquid and solid phases. It is an important mechanism of liquid metal embrittlement and corrosion [1–10]. Furthermore, investigations of the interfacial equilibrium at the intersection of the S/L interfaces with the GBs permit assessment of their relative energy as a function of temperature [21–23]. It was observed that the relative energy of the interfaces

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depends on the misorientation angle of the crystals separated by the GB [2, 8–17, 21–23] and is influenced by segregation [1, 12–14]. It was found that wetting of the GB by the liquid is related to diffusion of the low melting point elements along the GB [5, 18, 20]. For GBs in nickel wetted by liquid bismuth [5] and GBs in Fe-Si alloys wetted by liquid zinc [20] it is well established now that the wetting transition is preceded by the prewetting transition of the GBs. The prewetting transition is equivalent to the formation of a quasi-liquid film at the GB at temperatures below the melting point and at concentrations of the low melting point element where the bulk solid phase is stable. It is caused by diffusion of atoms of the low melting point metal along the GBs [20]. The concentration level of Zn at which the prewetting transition takes place is equivalent to the GB solubility limit of Zn [20]. The solubility limit of Zn in GBs in the Fe-Si alloys depends on the Si content, GB misorientation, temperature and pressure [20, 24–27, 29, 30]. The temperature and pressure effect on the Zn solubility limit was attributed to their effect on the Zn segregation coefficient [25, 26, 29, 30].

Investigations of the effect of pressure on GB wetting may provide important information about the nature of the wetting phenomena. However, there are only limited data on the pressure effect on the equilibrium between the S/L interface and GB [24–27]. Studies of the effect of pressure on GB wetting may permit estimation of the excess volumes of the S/L interface and GBs [28]. Furthermore, stress fields on the order of 1 GPa are frequent in technical applications, especially in thin films [31] and may strongly affect the GB wetting phenomena. Therefore, the pressure effect on GB wetting may have considerable technological importance.

Under equilibrium conditions the contact angle  $\theta$  at the intersection of the GB and the S/L interface (Fig. 1) is defined by the equation:

$$\cos\frac{\theta}{2} = \frac{\gamma_{\rm GB}}{2\gamma_{\rm SL}},\tag{1}$$

where  $\gamma_{GB}$  and  $\gamma_{SL}$  are the GB and S/L interface energy per unit area, respectively. Here torque terms [32–34], resulting from the inclination dependence of the surface energy, are neglected. We assume a linear dependence of the energy of both interfaces on pressure:

$$\frac{\theta}{2} = \arccos\left(\frac{\gamma_{\rm GBO} + pV_{\rm GB}}{2\gamma_{\rm SLO} + 2pV_{\rm SL}}\right),\tag{2}$$

where  $\gamma_{GBO}$  and  $\gamma_{SLO}$  are the energies of the GB and S/L interface at zero pressure, respectively, and V<sub>GB</sub>



*Figure 1.* Back-scattered electron image of the GB groove in the  $43^{\circ}$  (001) boundary after annealing at 905°C under a pressure of 0.75 GPa.

and  $V_{SL}$  are the excess volumes of these interfaces, and *p* is the pressure [23–26].

In the present paper we study the wetting of GBs by liquid zinc in Fe-6 at.% Si bicrystals. This is a reactive system and diffusion fluxes of Fe, Si and Zn perpendicular to the S/L interface take place. It is known from previous work [19, 20] that the Zn-rich melt wets the GBs completely. However, at pressures on the order of 0.5 GPa GB wetting is prevented and a dewetting transition takes place [24–27]. In a recent paper [27] we reported the results of investigations of the pressure and silicon concentration effect on the wetting angle in that system, a thermodynamic analysis of the equilibrium conditions at the interfaces and conditions for interfacial phase transitions. The purpose of the present paper is to propose a model of the S/L interface that will explain the observed strong effect of pressure on GB wetting.

Only two reports of the pressure effect on GB wetting in a system different from the Fe-Si alloy are known to the authors [28, 36]. Contrary to the present system, Stickels and Hucke [28], who studied the Ni-Pb system, observed no wetting transition. A moderate effect of the pressure on the wetting angle was noted, and the misorientation effect was not studied. At the same time the S/L interface between lead and nickel was not reactive, i.e., no diffusion fluxes perpendicular to the interface took place. Lee et al. [36] observed the dewetting transition in a ceramic material.

#### 2. Experimental

Three Fe-6% Si bicrystals were prepared by a technique described elsewhere [19]. Symmetrical tilt GBs with misorientation angles of  $35^\circ$ ,  $38^\circ$  and  $43^\circ$  and a  $\langle 001 \rangle$ misorientation axis were studied. The surface of the bicrystals perpendicular to the (001) axis was covered with a 100  $\mu$ m thick Zn layer. The high pressure experiments were carried out in a 99.999 wt.% argon atmosphere. All the bicrystals were wrapped in a tantalum foil and put in a stainless steel capsule of 9 mm internal diameter and 20 mm length, at the bottom of which about 50 mm<sup>3</sup> of Zn was deposited. The capsule was inserted into the high temperature furnace situated in the high pressure cell. The experiments were carried out as follows. First, the pressure was raised to a predetermined value and then the temperature was raised to 905°C. The temperature increase from 700°C to 905°C took place in 300 s. The final pressure resulted from the temperature increase at constant volume. The temperature was held constant for 360 s with a precision of  $\pm 1^{\circ}$ C by means of a temperature controller. The good heat conductivity of the compressed argon permitted cooling rates of about 1°C/s to be achieved. The pressure was released after cooling the samples to the room temperature. The wetting angle was determined by means of an optical microscope using a method described in [20] after cooling down the specimens. The concentration of Zn perpendicular to the S/L interface was measured by means of electron probe microanalysis. The concentration at the interface was taken as the limit of the concentration measured by EPMA when the probe approached the interface [27].

#### 3. Results and Discussion

At low pressures, the liquid Zn-rich film completely wetted all the GBs studied. At pressures in the range 0.4–0.6 GPa the dewetting transition had taken place. In that respect the behavior of the system studied was identical as reported in previous papers [24-27]. Figure 1 shows a scanning electron microscope image of the 43°GB grove after annealing at a pressure below and above the dewetting pressure. Table 1 shows the effect of the misorientation angle on the dewetting pressure. Figure 2(a)-(c) shows the pressure effect on the contact angle. The effect of pressure was not only to cause the dewetting transition. Above the dewetting transition pressure the contact angle increased with pressure. The change of the wetting angle was a function of the misorientation angle. For the  $35^{\circ}$  and  $43^{\circ}$ GBs, the wetting angle increased from  $0^{\circ}$ to about 100° above the dewetting pressure. On increasing the pressure further the change of  $\theta$  was about  $0.03^{\circ}$ /MPa. In the case of the near  $\Sigma 5$  GB the rate of the  $\theta$  change with pressure was about 0.15°/MPa, i.e., higher by a factor of 5. Figure 3 shows the pressure effect on the Zn concentration at the S/L interface for the sample with the  $35^{\circ}(001)$  GB. It is observed that there is a maximum of the Zn concentration at 0.8 GPa.

The present results confirm the previously reported strong pressure effect on the wetting of GBs in an Fe-6 at.% Si alloy by liquid Zn. Further, it was shown that the pressure effect on the wetting behavior of GBs depends on the misorientation angle.

Rabkin et al. [25] have shown that the pressure influence on GB wetting cannot be attributed to kinetic effects because the rate-controlling factor for the groove formation is the diffusion in the liquid, and the time for the formation of the GB groove is about 2500 s. It is two orders of magnitude longer than the time needed for the cooling or heating of the specimen. Since in the present experiments the pressure effect on GB wetting depends on the misorientation of the crystals, which does not influence the rate of the diffusional processes, the present experiments further confirm that the wetting phenomenon is governed by energetic factors.

*Table 1.* Summary of the results of the fitting procedure of Eq. (8) to the experimental data shown in Fig. 2. The lower limit for  $\Delta \gamma$  corresponds to the lower limit for  $\Delta V$  and the upper limit for  $\Delta \gamma$  to the upper limit for  $\Delta V$ , respectively.

Misorientation angle	Dewetting pressure (GPa)	$\partial  heta / \partial p$	$\Delta\gamma$ (mJ/m <sup>2</sup> )	$\Delta V$ (nm)
35°	$0.6\pm0.1$	0.03°/MPa	$400\pm100$	$0.65\pm0.15$
$38^{\circ}(\sim \Sigma 5)$	$0.8\pm0.1$	0.15°/MPa	$800\pm250$	$0.9\pm0.3$
43°	$0.4\pm0.1$	0.03°/MPa	$190\pm80$	$0.3\pm0.1$



*Figure 2.* Pressure effect on the wetting angle for  $\langle 001 \rangle$  boundaries having a misorientation angle of 35°(a), 38°(b) and 43°(c). The calculated curves correspond to the best fit values for  $\Delta \gamma$  and  $\Delta V$  to Eq. (2) according to Table 1.

Equation (2) shows that the pressure effect on GB wetting is controlled by the excess volume of the two interfaces. However, in a multicomponent system, changes of the excess volume may be connected with changes of the composition induced by the pressure. In fact, Fig. 3 shows that the pressure may cause changes



*Figure 3.* Effect of pressure on the Zn concentration at the S/L interface for the bicrystal with the  $35^{\circ}$  (001) GB.

of the chemical composition of the interfaces by a factor of 2. Therefore, a more thorough analysis of the meaning of the terms  $V_{SL}$  and  $V_{GB}$  in Eq. (2) is requested. In general, a change of the interfacial energy can be caused by both a change of pressure and composition:

$$d\gamma = \left(\frac{\partial\gamma}{\partial p}\right)_x dp + \left(\frac{\partial\gamma}{\partial x}\right)_p dx,\tag{3}$$

where *x* is the mole fraction of the alloy component. It is known that the anisotropy of the L/S interface energy is small and does not exceed 15% [31]. Therefore, it might be expected that the term  $(\partial \gamma / \partial x)_x$  does not depend on the orientation of the L/S interface and on the misorientation angle of the GBs. This is equivalent to our assumption that the term is identical for the three GBs studied.

Let us consider the  $(\delta \gamma / \delta p)_x$  term. In one-component systems, this term corresponds to the excess volume of the interface caused by the structural misfit of the two crystals or phases. In a multicomponent system, the GB excess volume may depend on the chemical composition as well. To assess this effect, we take into account that pressure may influence interfacial segregation and apply the concept of a segregation volume [29, 30, 35, 36]. We assume that on segregation of an atom of a given component A or B to the interface its atomic volume changes by the segregation volume  $V_A$  or  $V_B$ , respectively, compared to its volume in the bulk. Therefore, the excess volume of the interface is expressed as:

$$V = \frac{n_{\rm A} V_{\rm A} + n_{\rm B} V_{\rm B}}{n_{\rm A} + n_{\rm B}},\tag{4}$$

where  $n_A$  and  $n_B$  are the concentrations of the components A and B in the interface, respectively.

For a low mole fraction of  $B: x_B = n_A/(n_A + n_B) \ll 1$ , Eq. (4) reduces to:

$$\frac{\partial \gamma}{\partial p} = V_{\rm A} + x_{\rm B}(p)V_{\rm B}.$$
(5)

The pressure effect on the segregation factor can be expressed as [29, 30, 35, 36]

$$x_{\rm B}(p) = x_{\rm B}^0 \exp\left(\frac{-pV_B}{kT}\right) \approx x_{\rm B}^0 \left(1 - \frac{pV_{\rm B}}{kT}\right), \quad (6)$$

where  $x_B^0$  is the mole fraction of B atoms in the interface at zero pressure. Substituting Eq. (6) into Eq. (5) and neglecting the  $x_B^2$  terms, we finally get the following expression for the effective excess volume for a binary system:

$$V = V_{\rm A} + x_{\rm B}^0 V_{\rm B}. \tag{7}$$

The equation for a three-component system can be derived in a similar manner. It follows that in the present experiments the excess volume of the interface depends on the average excess volume per atom of three components Zn, Si and Fe.

During the fitting procedure of the experimentally determined  $\theta(p)$  dependencies by the Eq. (2) we used the following values:  $\gamma_{\text{GBO}} = 800 \pm 200 \text{ mJ/m}^2$  and  $\gamma_{\text{SL}} = 300 \text{ mJ/m}^2$  [32–34, 37]. Owing to the irregular shape of the GB groove, the error of the wetting angle determination is about  $\pm 10^\circ$ . By fitting Eq. (2) to the experimental data it was found that the results weakly depend on the absolute values of interfacial excess volumes and energies. However, the shape of the fitting curve is sensitive to the differences:

 $\Delta V = 2V_{\rm SL} - V_{\rm GB}$ 

$$\Delta \gamma = \gamma_{\rm GB} - 2\gamma_{\rm SL}.\tag{9}$$

(8)

Therefore, fixed values  $V_{GB} = 0$  nm and  $2\gamma_{SL} = 600 \text{ mJ/m}^2$  were assumed and the above differences were treated as fitting parameters. Neglecting the GB excess volume is justified in the light of recent calculations by Wolf and Merkle [38] who have shown that it is less than 0.04 nm, i.e., it is small comparing to the S/L interface excess volume measured in the present work. The excess volume is expressed in terms of

the thickness of a slice of vacuum, which would cause the same change of the density of the bicrystal as the given GB. Using this procedure, the lower limit for  $V_{SL} = \Delta V$  can be estimated. This is assumed to be the main result of the fitting procedure.

The calculated  $V_{SL}$  are summarized in Table 1. It can be seen that for all the bicrystals studied the  $V_{SL}$  value is higher than 0.2 nm. Table 1 shows the two main results of the present paper:

- (a) The S/L interface is characterized by a high excess volume.
- (b) The dewetting pressure is higher for the near  $\Sigma 5$  GB than in the case of the other two GBs studied.

It is well known from thermodynamic considerations [39–41] and modeling studies [38–42] that in the case of a low heat of fusion, the S/L interface is diffuse and assumes a thickness of a few atomic diameters. This conclusion was confirmed by direct observations using high-resolution transmission electron microscopy [43]. The excess volume of the S/L interface was attributed to the thin layer between the solid and liquid where the arrangement of atoms is neither the same as in the liquid phase nor as in the solid phase. Straumal et al. [27] proposed to take into account in addition the misfit at the S/L interface caused by differences in size of the atoms in the solid and melt. However, these factors contribute to the excess volume by a factor on the order of magnitude of the GB excess volume or less [39-46]. The GB excess volume, according to the computer calculations of Wolf and Merke [38] is one order of magnitude less than the S/L interface excess volume found in the present paper. To explain the high S/L interface excess volumes observed in the present work it is necessary to assume a one order of magnitude thicker S/L interface than estimated in [39–46].

The interface thickness L, the excess volume  $V_{SL}$ , and the density difference between the solid and liquid phases  $\Delta \rho$  can be related by the equation (cf. Fig. 5):

$$L\Delta\rho = V_{\rm SL}.\tag{10}$$

We assume that the density deficit of the S/L interface originates from the fact that the liquid is less dense than the solid. In other words, the presence of the interface prevents the liquid from crystallizing and contributes to a change in the whole volume of the system. Figure 5 illustrates the above concept of the excess volume for the S/L interface. If the interface width L decreases to zero, the equivalent volume of the liquid, represented



*Figure 4.* Two possible explanations of the high dewetting pressure for the near  $\Sigma 5$  GB.  $P_1$  corresponds to the dewetting pressure for a general GB. It takes place when the energy of two S/L interfaces is higher than the GB energy—point (a).  $P_2$  corresponds to the dewetting pressure for the near  $\Sigma 5$  GB. It is higher either because the two S/L surfaces parallel to the symmetrical near  $\Sigma 5$  GB (310) have a small excess volume—point (b), or the near  $\Sigma 5$  GB has a high energy—point (c).



*Figure 5.* Illustration of the concept of the excess volume for the "rough" S/L interface. It is assumed that the S/L interface has a liquid-like structure with solid-like clusters (represented by density oscillations). The width of the interface corresponds to the range of the existence of the clusters. We neglect the effect of the clusters on the density. If the interface width L decreases to zero, the equivalent volume of the liquid, represented by the shadowed area, is replaced by the solid. As a consequence, the volume of the system decreases by  $V_{\rm SL} = L \Delta \rho$ . The absolute value of the change of volume per unit surface is defined as the excess volume of the S/L interface.

by the shadowed area, is replaced by the solid, and the volume of the system decreases by  $V_{SL} = L\Delta\rho$ . Since the density difference between the liquid and solid is on the order of 2% and the minimum value of  $V_{SL}$  found

was 0.2 nm, Eq. (10) leads to the result that the S/L interface thickness L is on the order of 10 nm.

The above result indicates long-range interaction forces between the two crystals separated by a thin liquid film. Such long-range interaction forces cannot be excluded in light of recent experimental results concerning the wetting of GBs by liquid metal [16, 17]. We propose an explanation in terms of the association model of liquid alloys [47] of the long-range interaction forces between two crystals separated by the melt. Let us assume that the S/L interface has a liquid-like structure with solid-like clusters (represented in Fig. 5 by density oscillations). The width of the interface corresponds to the equilibrium range of existence of the clusters. In the present case the S/L interface is at the same time an interface between the Fe-rich solid and Zn-rich liquid takes place. So it is natural to assume that the presence of the intermetallic  $\Gamma$  phase in the solid contributes to the stability of Fe-Zn clusters in the liquid.

As far as the near  $\Sigma 5$  GB is concerned, the fitting procedure could not lead to reasonable values of the fitting parameters (Fig. 2(b)). Figure 4 shows that there are two possible explanations for the high dewetting pressure. The first explanation is that the near  $\Sigma 5$  GB possesses a higher energy than the general GBs. The second explanation is that even in the wetted state this boundary has a low excess volume. As far as the first explanation is concerned, although the  $\Sigma 5$  GB is known to be connected with an energy minimum in pure metals, this need not to be the case in an alloy. The data for the effect of misorientation on the interface energy in alloys are scarce and the low energy GBs for alloys need not to be the same as for pure metals. However, it seems unlikely that the near  $\Sigma 5$  has a significantly higher energy than the general GBs. Except for some special (011) tilt GBs energy differences between special and general GBs are on the order of a few percent [38, 48, 49]. Such a small energy increase could not explain the observed differences in the dewetting pressure between the GBs investigated. Therefore the second possibility seems more plausible. It means that the liquid film wetting the near  $\Sigma 5$  boundary is thinner than for the case of a general boundary. Such a situation would indicate that the two [310] planes of the  $\Sigma 5$  interact across the liquid film and even in the wetted state this GB is not completely disordered. A low excess volume of the wetted  $\Sigma 5$  GB would explain the high dewetting pressure for this boundary. However, to obtain the fitting parameters  $\Delta \gamma$  and  $\Delta V$  it is necessary to fit Eq. (2) to all the data points not only to the dewetting pressure. As can be seen from Fig. 2(b), fitting of Eq. (2) to the experimental points of the near  $\Sigma 5$  GB was not possible. The singular properties of the near  $\Sigma 38^{\circ} \langle 001 \rangle$  GB cannot be fully explained in the framework of the present model.

The high excess volume of the S/L interfaces found in the present study is in contrast with the low excess volumes found by Stickels and Hucke [28] in the Ni-Pb system. However, these authors studied a system in thermodynamic equilibrium, where no interdiffusion perpendicular to the interface took place. In contrast, in the present study a continuous dissolution process of the Fe and Si atoms into the Zn-rich melt proceeds. At the same time Zn diffused into the bulk. It might be suggested that the present interface is rough, with channels of liquid Zn penetrating into the bulk and clusters of atoms detaching from the solid and moving into the liquid. Such a process increases the S/L contact surface and the dissolution process is accelerated. The presence of such clusters may contribute to the long-range repelling force between the two crystals. Assuming a cluster size of about 5 atoms, an interface thickness of about 10 nm would correspond to about one to five clusters in the transition zone. The above model of the reactive S/L interface shows some similarities to the sea-iceberg interfacial zone, where icebergs detach from the bulk ice, drift towards the open sea and slowly begin to melt. Therefore, it might be called "melting iceberg model for the solid/liquid interface", which should be applicable to reactive systems like the present Fe-6 at.% Si alloy wetted by liquid Zn.

#### 4. Conclusions

From the present studies the following conclusions can be drawn.

- 1. The strong pressure effect on the grain boundary wetting of Fe-6 at.%Si bicrystals by liquid Zn indicates a high excess volume of the solid/liquid interface of more than 0.2 nm. The high excess volume of the solid/liquid interface leads to a dewetting transition as the pressure is increased.
- 2. The high excess volume indicates a diffuse character of the interface.
- The present results speak for long-range interactions
   (≈10 nm) between the crystals across the liquid. To
   explain such a long-range interaction, the following
   concept was proposed: solid-like clusters detach
   from the crystal and liquid-like Zn-rich channels
   penetrate into the solid. The interaction distance is
   equal to the distance at which the clusters dissolve

in the melt. The above model for the solid/liquid interface is called the "melting iceberg model" for the solid/liquid interface.

4. The dewetting transition pressure is higher for the near  $\Sigma 5$  special grain boundary than for the two general boundaries studied. This result was interpreted as evidence for thinner liquid film between two S/L surfaces of equal and short periodicity comparing to the nonperiodic GBs.

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