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Effect of the Temperature Gradient, Growth Rate, and the Interflake Spacing on the Microhardness in the Directionally Solidified Al-Si Eutectic Alloy

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An alloy of composition Al-12.6 wt.% Si was prepared using metals of 99.99% purity. Weighed amounts of aluminium and silicon were melted in the vacuum-melting furnace. This irregular eutectic alloys were directionally solidified upward with a constant growth rate V (8.3×10^{-3} mm/s) and different temperature gradients G (2.0-7.8 K/mm) and also with a constant temperature gradient G (7.8 K/mm) and different growth rates V ($8.3\text{-}498.7 \times 10^{-3}$ mm/s) in the directional solidification furnace. The interflake spacings λ and microhardness H_V were measured from both transverse section and longitudinal section of the specimen. The variations of H_V with respect to G, V, and λ have been determined by using the linear regression analysis method. It has been shown that H_V increases with the increasing values of G and V. On the other hand H_V values decreases with the increasing λ values. The Hall-Petch type relationships obtained in this work have been compared with the previous works.

Keywords Al-Si eutectic alloy, Hall-Petch relationships, interflake spacings, microhardness

1. Introduction

Al-Si eutectic alloys are one of the most widely used aluminium foundry alloys today. Refining the eutectic silicon morphology by modification has been used extensively industrially since about the 1970s to improve the mechanical properties of the casting.[1] Consequently, eutectic crystals show excellent mechanical properties at room temperature and good retention of these properties up to temperatures near the eutectic point. It is known that the mechanical properties of metallic materials are affected by theirs morphology. The mechanical properties of directional solidification Al-Si eutectic, which is an important commercial material have been reported in several investigation,[2-4] but the results differ from each other. In this work, we are reporting microhardness properties of directional solidification eutectic grown at different temperature gradients or different growth rates.

The solidification of eutectic alloys generally gives rise to flake, fibrous, or complex regular structures. The spacing of the interflake or fibrous is typically regular with a dispersion around an average value.

The purpose of the present work is to experimentally investigate the dependence of the, temperature gradient, G, growth rate, V, and interflake spacing, λ, on the microhardness, H_V. Al-Si eutectic systems have been chosen for the study due to their eutectic structure, widely available experimental results, and well-defined physical properties. The detailed analysis between the average interflake spacing λ and the solidification parameters G and V for Al-Si has been given in Ref. 5.

Several studies[6-9] have related structure and mechanical properties in directionally solidified different alloy systems and analyzed their mechanical properties in terms of composite behavior. Many studies[8-15] of Al-Si eutectic alloys show that hardness and growth rate are related by an equation of the form:

$$H_V = H_o + K_1 V^a \qquad \text{(Eq 1)}$$

Many researchers[6,8-17] proposed that the dependence of hardness and interflake spacings (grain size) are related by an equation of the Hall-Petch[6,7] type of behavior described by:

$$H_V = H_o + K_2 \lambda^{-b} \qquad \text{(Eq 2)}$$

where H_V is the hardness, H_o is the initial hardness, λ is interflake spacing, V is growth velocity, and K_1 and K_2 are constant. This relationship has been shown to be valid for Cu alloys,[17] steel alloys,[18] Zn alloys,[19] Pb-Cd eutectic alloy,[20] and Cu, Ag, Al alloys.[21]

Theoretical and experimental investigations by many researchers have revealed various eutectic systems.[1,22-25]

The experimental results will be presented first and compared with current results in the literature for eutectic alloys to get more general information about the relationships between H_V and V and λ.

2. Experimental Procedure

An alloy of composition Al-12.6 wt.% Si was prepared using metals of 99.99% purity. Weighed amounts of aluminium and silicon were melted under vacuum melting furnace. After

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allowing time for melt homogenization, molten alloy was poured into the prepared 13 graphite crucibles (250 mm in length, 4 mm inner diameter (ID), 6.35 mm outer diameter (OD) in a hot filling furnace. Then each specimen was positioned in a Bridgman-type furnace in a graphite cylinder (300 mm in length, 10 mm ID, 40 mm OD). Accuracy of the thermocouples was checked by slowly solidifying the alloy system (which were thermocouples placed parallel to the heat flow and perpendicular to the heat flow direction). The measured eutectic temperature, T_E (850.4 K) difference was less than 0.5 K with differently placed thermocouples. The temperature of the Bridgman-type furnace was controlled by a Pt/Pt-13%Rh thermocouple placed between the heating element and the alumina tube. The temperature could be controlled to about ±0.1 K during the run. The thermocouples were placed into the capillary alumina tubes (0.8 mm ID, 1.2 mm OD) which were positioned approximately 10 mm apart and parallel to the heat flow direction inside the crucible. Throughout the experiment, temperature distribution was obtained by measuring the temperature in the sample by three chromel/alumel thermocouples (type-K), which were placed in the sample. All the thermocouple leads were taken to an ice/water cold junction, then to a WPA analog potentiometer and to a Kipp-Zonen chart recorder capable of recording to 1 μV. After stabilizing the thermal conditions in the furnace under an argon atmosphere, the specimen was grown by pulling it downwards at various constant rates by means of different speed synchronous motors. Specimens were solidified under steady state conditions with a constant growth rates V (8.3×10^{-3} mm/s) and different temperature gradients G (7.8 K/mm) and also with a constant temperature gradient, G (7.8 K/mm) and different growth rates, V ($8.3\text{-}498.7 \times 10^{-3}$ mm/s) in the directional solidification furnace (Table 1). After 100-120 mm steady state growth of the samples, they were quenched by pulling them rapidly into the water reservoir.

2.1 Metallographic Examination

The unidirectionally grown quenched specimens were removed from the graphite crucible and 3 cm lengths from the top and bottom were cropped off and discarded, then ground to observe the solid-liquid interface and the longitudinal section,

Table 1a The Experimental Relationships Among Microhardness, Growth Rate, and Interflake Spacing at the Constant Growth Rate in the Directionally Solidified Al-Si Eutectic Alloy System

Al-Si Eutectic Alloy System (Different Temperature Gradient, Constant Growth Rate)

Solidification Parameters		Interflake Spacings		Microhardness		
G, K/mm	$V \times 10^{-3}$, mm/s	$\lambda^* \times 10^{-3}$, mm	$\lambda^{**} \times 10^{-3}$, mm	H_V^*, kg/mm^2	H_V^{**}, kg/mm^2	Relationships
2.0	8.3	14.2 ± 0.3	13.6 ± 0.5	52.6 ± 0.7	53.2 ± 0.6	$H_V^* = k_1 G^{0.08}$
3.2	8.3	13.2 ± 0.1	12.6 ± 0.2	54.3 ± 0.7	55.1 ± 1.7	$H_V^{**} = k_2 G^{0.09}$
4.4	8.3	12.3 ± 0.2	11.2 ± 0.4	55.9 ± 1.0	56.3 ± 0.8	$H_V^* = k_3(\lambda^*)^{-0.15}$
5.7	8.3	11.2 ± 0.2	10.1 ± 0.2	57.0 ± 1.1	58.7 ± 1.8	$H_V^{**} = k_4(\lambda^{**})^{-0.17}$
						…
7.8	8.3	10.7 ± 0.2	8.2 ± 0.2	59.1 ± 1.1	60.0 ± 2.0	

Constant (k)	Correlation Coefficients (r)
$k_1 = 49.28(K^{-0.08} \cdot kg \cdot mm^{-1.92})$	$r_1 = 0.989$
$k_2 = 49.54(K^{-0.09} \cdot kg \cdot mm^{-1.91})$	$r_2 = 0.994$
$k_3 = 27.54(kg \cdot mm^{-1.82})$	$r_3 = -0.985$
$k_4 = 27.54(kg \cdot mm^{-1.81})$	$r_4 = -0.991$

Table 1b The Experimental Relationships Among Microhardness, Growth Rate, and Interflake Spacing at the Constant Temperature Gradient in the Directionally Solidified Al-Si Eutectic Alloy System

Al-Si Eutectic Alloy System (Different Growth Rate, Constant Temperature Gradient)

Solidification Parameters		Interflake Spacings		Microhardness		
G, K/mm	$V \times 10^{-3}$, mm/s	$\lambda^* \times 10^{-3}$, mm	$\lambda^{**} \times 10^{-3}$, mm	H_V^*, kg/mm^2	H_V^{**}, kg/mm^2	Relationships
7.8	8.3	10.7 ± 0.2	8.2 ± 0.2	59.1 ± 1.1	60.0 ± 2.0	$H_V^* = k_5 V^{0.09}$
7.8	16.4	9.6 ± 0.1	7.3 ± 0.2	62.5 ± 0.9	62.0 ± 1.8	$H_V^{**} = k_6 V^{0.11}$
7.8	41.0	6.7 ± 0.2	4.5 ± 0.4	65.5 ± 0.9	65.0 ± 1.0	$H_V^* = k_7(\lambda^*)^{-0.16}$
7.8	82.4	3.8 ± 0.1	3.0 ± 0.3	69.1 ± 0.4	69.0 ± 0.5	$H_V^{**} = k_8(\lambda^{**})^{-0.18}$
7.8	165.2	2.0 ± 0.1	2.1 ± 0.4	74.2 ± 1.9	73.4 ± 1.4	…
						…
7.8	498.7	1.8 ± 0.1	1.4 ± 0.1	81.4 ± 0.4	79.5 ± 1.1	

Constant, k	Correlation Coefficients (r)
$k_5 = 84.92(kg \cdot mm^{-2.09} \cdot s^{0.09})$	$r_5 = -0.994$
$k_6 = 82.79(kg \cdot mm^{-2.11} \cdot s^{0.11})$	$r_6 = -0.991$
$k_7 = 25.35(kg \cdot mm^{-1.84})$	$r_7 = -0.988$
$k_8 = 27.54(kg \cdot mm^{-1.82})$	$r_8 = -0.997$

which included the quenched interface was separated from the specimen. This part was ground, polished, and etched to reveal the quenched interface. Furthermore, the longitudinal and the transverse sections of the ground specimen were mounted in a cold-setting epoxy resin. The microstructural of the specimens were determined by metallographic analysis. Mechanical and electropolishing techniques were used to prepare the transverse and the longitudinal sections for both optical microscopy (OM) and scanning electron microscopy (SEM) (Fig. 1 and 2).

2.2 Measurement of Growth Rates, V, and Temperature Gradient, G

The thermocouples were recorded simultaneously for measurement of the growth rates and the temperature gradients on the solid-liquid interface in the liquid. When the second thermocouple was at the solid-liquid interface and the third thermocouple was in the liquid their temperatures were used to obtain the temperature gradient, G. The values for the growth rates, V, were calculated from the measurements of the time taken for the solid-liquid interface to pass the thermocouples separated by a known distance. The position of the thermocouples was measured after the quench. The values of G and V are given in Table 1(a) and (b). The experimental details are given in Ref. 26.

2.3 Measurement of Interflake Spacing, λ, and Microhardness, H_V

The samples were prepared for microstructural examination, including the solid-liquid interface on the longitudinal section. The transverse section was taken near the solid-liquid interface (2-3 mm) to measure λ. The average interflake spacings, λ^* values, were measured on the longitudinal section and λ^{**} values were measured on the transverse section of the samples (Fig. 1 and 2). Interflake spacings λ^* and λ^{**} were measured at least in 4-6 different regions on the longitudinal section and 8-10 different regions on the transverse section. A linear intercept method was used to measure average eutectic

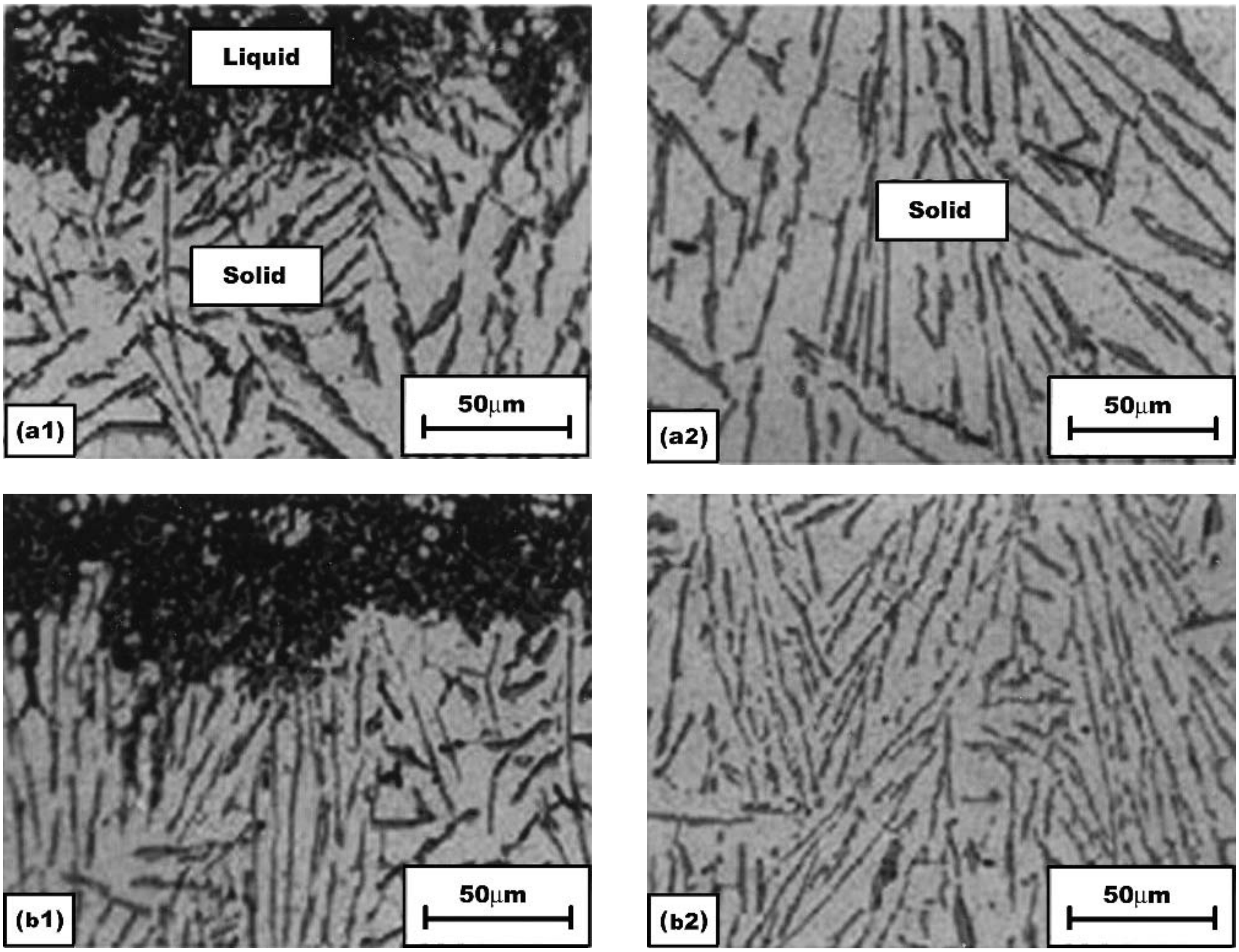

Fig. 1 Optical and SEM micrographs of the directional solidified Al-Si eutectic showing the flake structure, at a constant V (8.3×10^{-3} mm/s) and different G: **(a_1)** longitudinal section; **(a_2)** transverse section (G = 2.0 K/mm); **(b_1)** longitudinal section; **(b_2)** transverse section (G = 7.8 K/mm). (Continued on next page)

flake spacing longitudinal and transverse sections. Interflake spacings was measured from photographs (Fig. 1 and 2) of longitudinal and transverse sections of magnification, M, by counting the number of flake, N, within a known area, A. The interflake spacing was calculated from Ref. 27:

$$\lambda = \frac{1}{M}\left(\frac{A}{N}\right)^{0.5} \qquad \text{(Eq 3)}$$

Microhardness values (H_V) of the specimens for Al-Si eutectic system were also measured at the same places where λ was measured and on the transverse section and the longitudinal section, using a Vickers type Highwood model microhardness measuring test device equipped with a square-based pyramidal indenter with an angle of 136°. Ten indentations were obtained from each specimen using the test loads of 10-25 gf were used at these microhardness analysis. The microhardness values were the average of at least 10 measurements on longitudinal section (H_V*) and transverse section (H_V**). The minimum impression spacing (center to edge of adjacent impression) was about 3 times the diagonal and was located at least 0.5 mm from the edge of the specimen. G, V, λ, and H_V values are also given in Table 1(a) and (b).

3. Result and Discussion

Al-Si eutectic samples were directionally solidified with a constant growth rate V (8.3×10^{-3} mm/s) and different temperature gradients G (2.0-7.8 K/mm) and also with a constant temperature gradient G (7.8 K/mm) and different growth rates V ($8.3\text{-}498.7 \times 10^{-3}$ mm/s) to see the effect of the temperature gradient and the growth rate on the interflake spacings (λ*, λ**) and microhardness (H_V*, H_V**). As can be seen from Fig. 1 and 2 during eutectic growth, a large number of eutectic grains can be formed. All grains seem to have different growth orientation. Interflake spacings (λ*and λ**) and microhardness (H_V*and H_V**) measurements made on flake structures of longitudinal and transverse sections were recorded (Table 1).

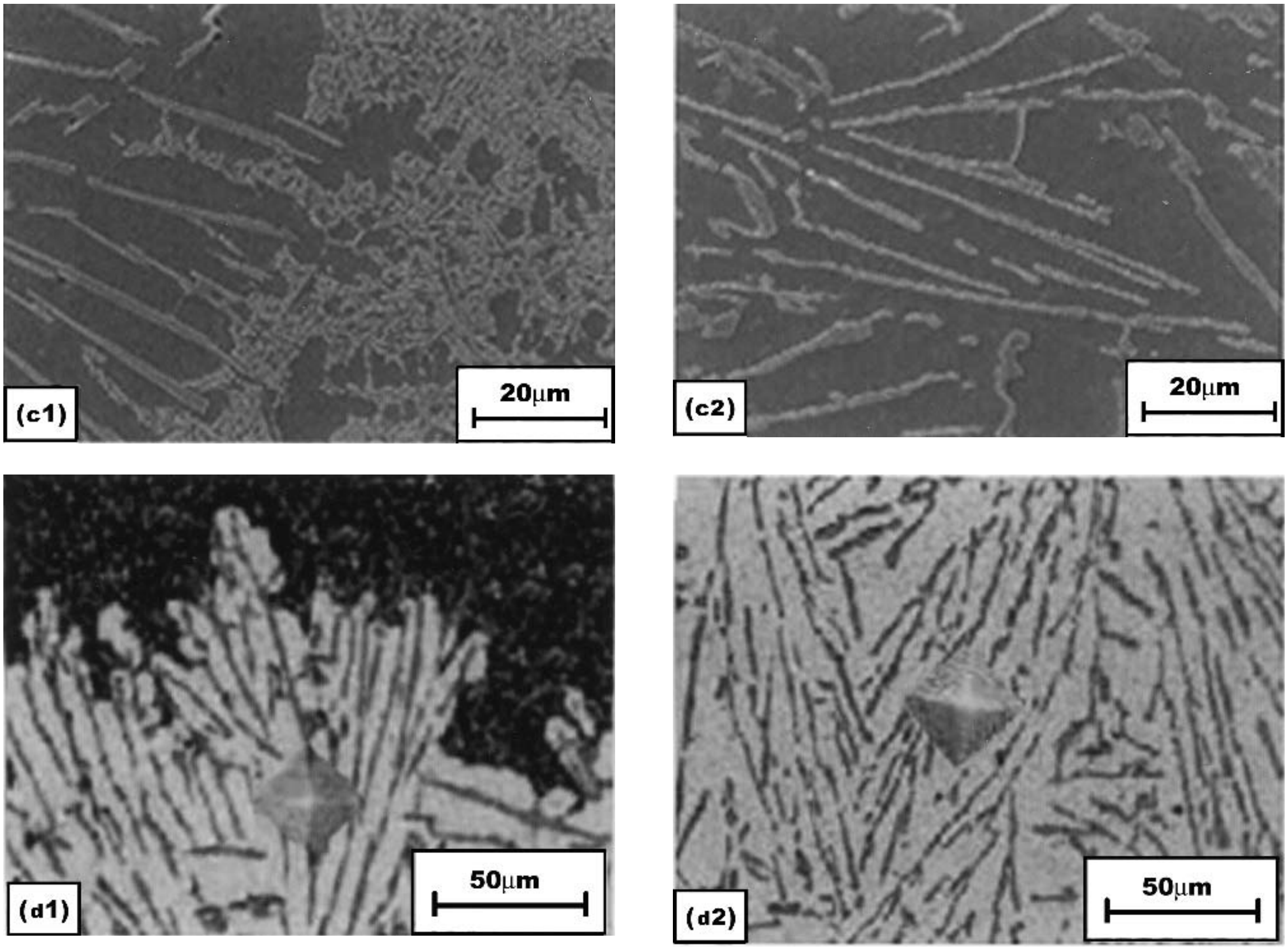

Fig. 1 cont. Optical and SEM micrographs of the directional solidified Al-Si eutectic showing the flake structure, at a constant V (8.3×10^{-3} mm/s) and different G: **(c_1)** longitudinal section (SEM); **(c_2)** transverse section (SEM, G = 2.0 K/mm); **(d_1)** the trace of square-based pyramidal indenter on directionally solidified Al-Si eutectic in microhardness measurements (longitudinal section, G = 4.4 K/mm, V = 8.3×10^{-3} mm/s); and **(d_2)** transverse section.

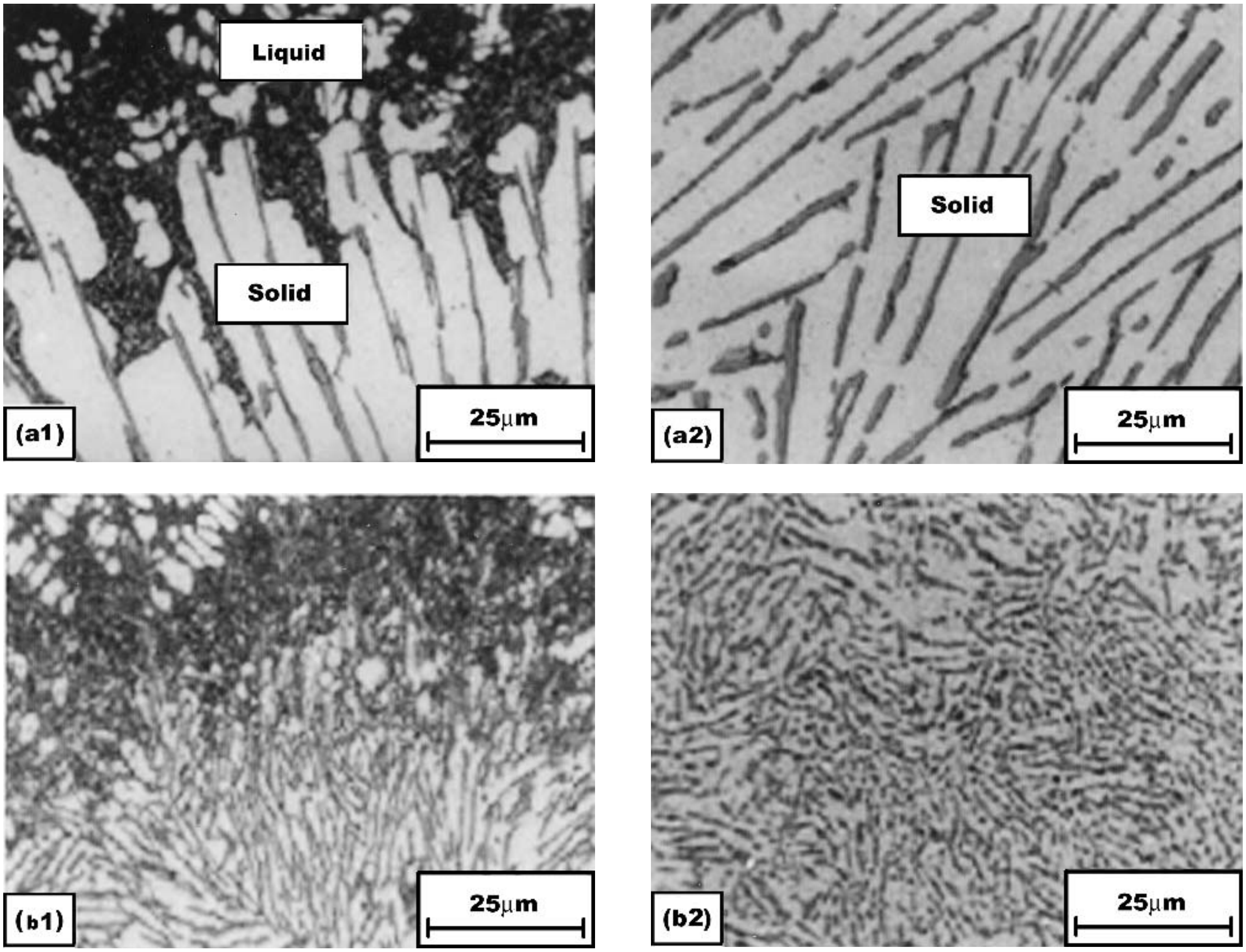

Fig. 2 Optical and SEM micrographs of the directional solidified Al-Si eutectic showing the flake structure, at a constant G (7.8 K/mm) and different V: **(a_1)** (longitudinal section) **(a_2)** transverse section ($V = 8.33 \times 10^{-3}$ mm/s); **(b_1)** longitudinal section; **(b_2)** transverse section ($V = 598.7 \times 10^{-3}$ mm/s). (Continued on next page)

3.1 Relationships Between the Microhardness and the Temperature Gradient

The influence of G cannot be ignored for regular or irregular eutectic systems. The influence of temperature gradient on the lamellar or interflake spacings was investigated by several authors.[28-31] An increase in the temperature gradient leads to an increase in the microhardness (H_V* and H_V**) for a given constant growth rate as well (Table 1a). The variation of the temperature gradient, G, as a function of the temperature gradients is given in Fig. 3(a).

As can be seen from Fig. 3(a) and Table 1(a), H_V* and H_V** increase with the increasing G for a constant V. Thus we can describe the mathematical relationship between H_V and G by linear regression analysis as:

$$H_V = k_1 G^a \quad \text{(for the constant V)} \qquad \text{(Eq 4)}$$

where k_1 is proportionality constant and given in Table 1(a). The dependence of the microhardness (H_V* and H_V**) on the temperature gradient exponent is equal to 0.08 and 0.09 for longitudinal section and transverse section, respectively (Table 1a and Fig. 3a). These values are slightly greater than previous measurements for alloys solidified at different temperature gradients,[10,32] c = 0.04.

3.2 Relationships Between the Microhardness and the Growth Rate

Variation of H_V as a function of V at the constant G is shown in Fig. 3(b) and Table 1(b) for Al-Si eutectic system. A linear regression analysis gives the proportionality equation as:

$$H_V = k_2 V^b \quad \text{(for the constant G)} \qquad \text{(Eq 5)}$$

Figure 3(b) and Table 1(b) clearly show that an increase in growth rate, V, produces an increase in microhardness, H_V. The value of the exponent relating to growth rate, b, is equal to

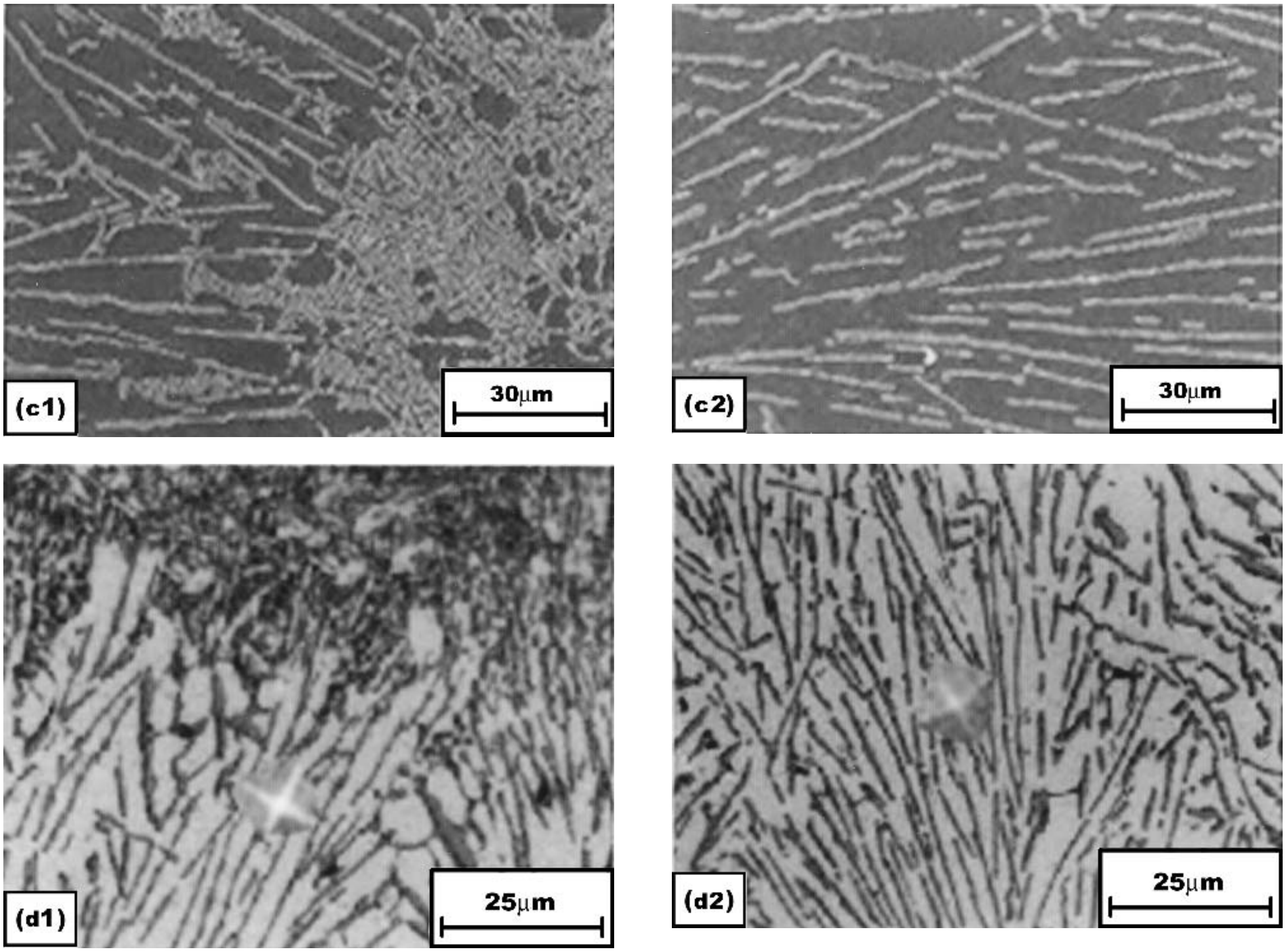

Fig. 2 cont. Optical and SEM micrographs of the directional solidified Al-Si eutectic showing the flake structure, at a constant G (7.8 K/mm) and different V: **(c_1)** longitudinal section (SEM); **(c_2)** transverse section (SEM, $V = 41.0 \times 10^{-3}$ mm/s); **(d_1)** the trace of square-based pyramidal indenter on directionally solidified Al-Si eutectic in microhardness measurements (longitudinal section, G = 7.8 K/mm, $V = 165.2 \times 10^{-3}$ mm/s); and **(d_2)** transverse section.

0.09 and 0.11 for longitudinal section and transverse section, respectively.

The exponent value, b = 0.11 for transverse section has been compared with previous results[8,10,12-14] for similar solidification conditions in Al-Si eutectic alloys. The exponent value in this work 0.11 is fairly close to the 0.12 value obtained by Khan et al.[14] and the 0.08 value obtained by Telli and Kısakürek[12] for Al-Si eutectic alloy, but slightly higher than the values (0.04 and 0.034) obtained by Yılmaz and Elliott[10] and Yılmaz,[13] respectively.

3.3 Relationships Between the Microhardness and the Interflake Spacing

Variation in H_V with λ is shown in Fig. 4(a) and (b) for Al-Si eutectic alloy. A linear regression analysis gives the proportionality of the Hall-Petch type equation as:

$$H_V = k\lambda^{-c} \qquad \text{(Eq 6)}$$

As can be seen from Fig. 4 and Table 1(a) and (b), an increase in λ produces a decrease in H_V. The value of the exponent relating to interflake spacing, c is equal to 0.15 and 0.17 for longitudinal and transverse section, respectively, for the constant growth rate, V. Also, the value of the exponent relating to interflake spacing, c is equal to 0.16 and 0.18 for longitudinal and transverse section, respectively, for the temperature gradient, G. The exponent value of c, 0.18, for transverse section is in good agreement with the 0.22 value obtained by Khan et al.,[14] but slightly higher than 0.08 value obtained by Yılmaz and Elliott.[10]

As seen in Fig. 5(a) and (b), the experimental H_V values for Al-Si eutectic alloys are in good agreement with values of the available literature under the similar solidification conditions. Figure 5(a) shows the variation of microhardness, H_V, as function of growth rate, V. H_V increases with the increasing V. Also Fig. 5(b) shows the variation of microhardness, H_V, as a function of lamellar spacing, λ. H_V increases with the decreasing λ.

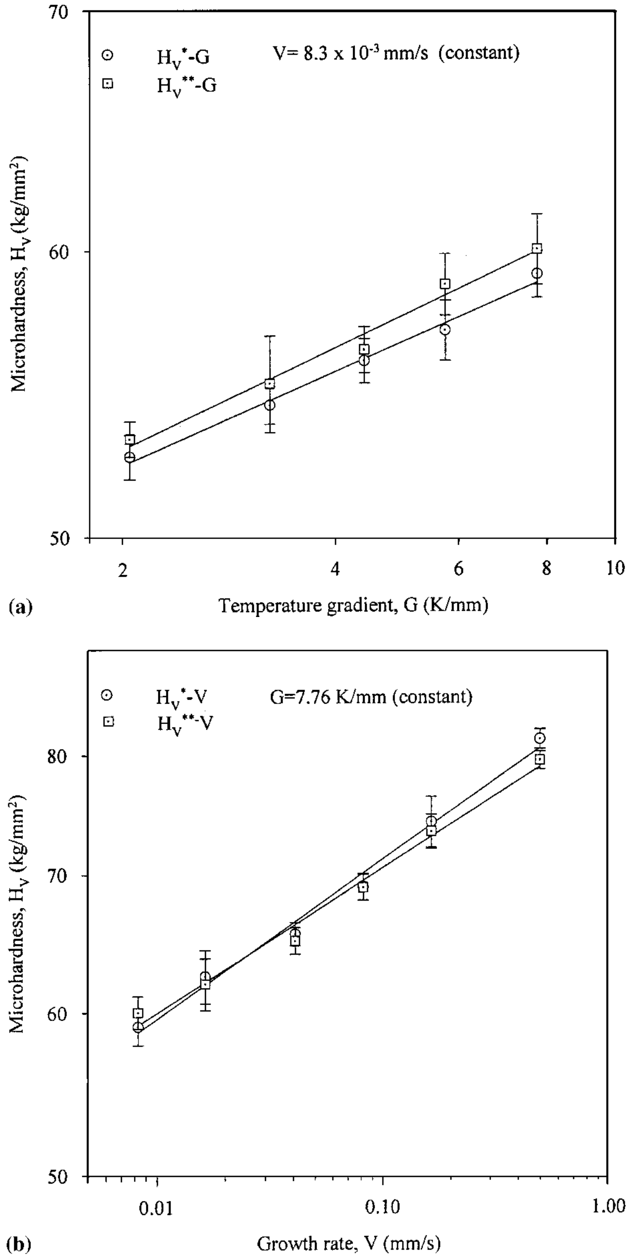

Fig. 3 **(a)** Variation of microhardness H_V as a function temperature gradient, G at a constant growth rate, V (8.33 x 10^{-3} mm/s) for Al-Si eutectic alloy; **(b)** variation of microhardness H_V as a function growth rate V, at a constant temperature gradient (G = 7.8 K/mm).

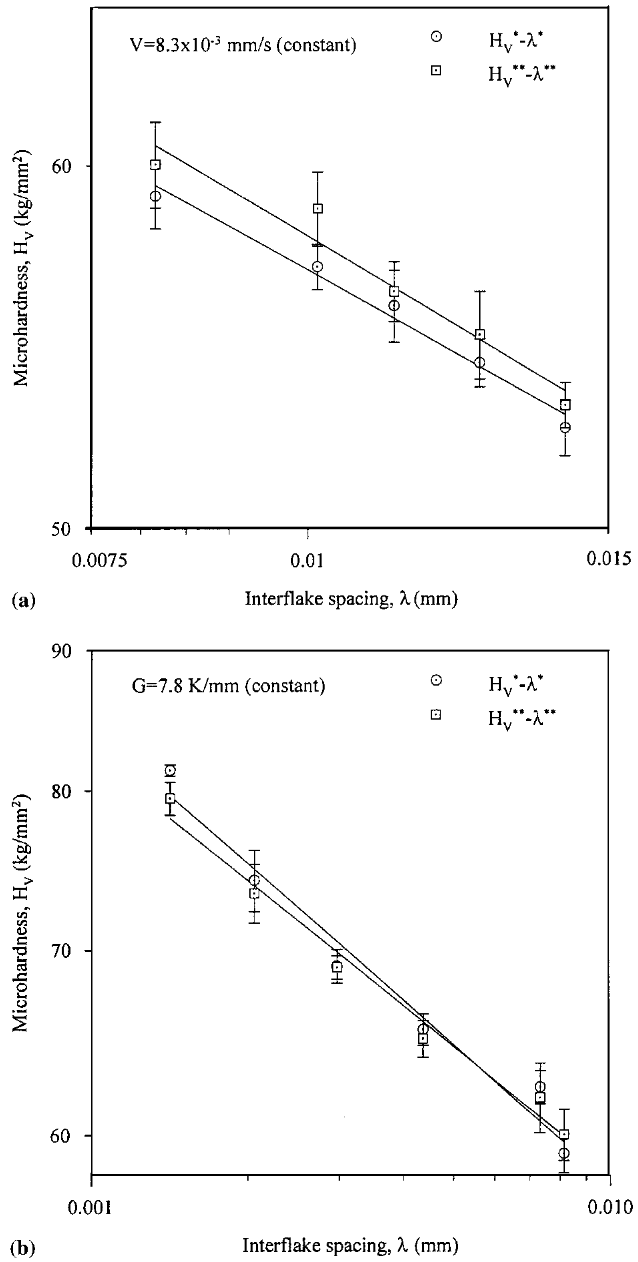

Fig. 4 **(a)** Variation of microhardness H_V as a function lamellar spacing, λ for a constant growth rate; **(b)** variation of microhardness H_V as a function lamellar spacing, λ for a constant temperature gradient.

4. Conclusion

- The microhardness values of the specimens, H_V, were measured in at least 10 regions on the transverse and longitudinal sections. It was found that the hardness values (H_V) of the specimens increased as G values were increased. The relationships between H_V and G can be given as $H_V = kG^a$.
- H_V values increased as V values were increased. The relationships between H_V and V can be given as $H_V = kV^b$. The value of *a* is 0.09 for transverse section. The exponent value b is 0.11 for transverse section and in good agreement with other workers results.[8,10,12-14]
- The relationships between the microhardness and the interflake spacing were obtained by linear regression analysis. It shows that the value of H_V increases as the values λ decrease. The establishment of the Hall-Petch type relationships given as $H_V = k\lambda^{-c}$ relating to these parameters. The exponent value, c, is 0.18 for transverse section and in good agreement with other workers results.[8,10,12-14]
- Dependence of H_V on V and λ found in this work for Al-Si eutectic alloy in good agreement with the previous results.[8,10,12-14]

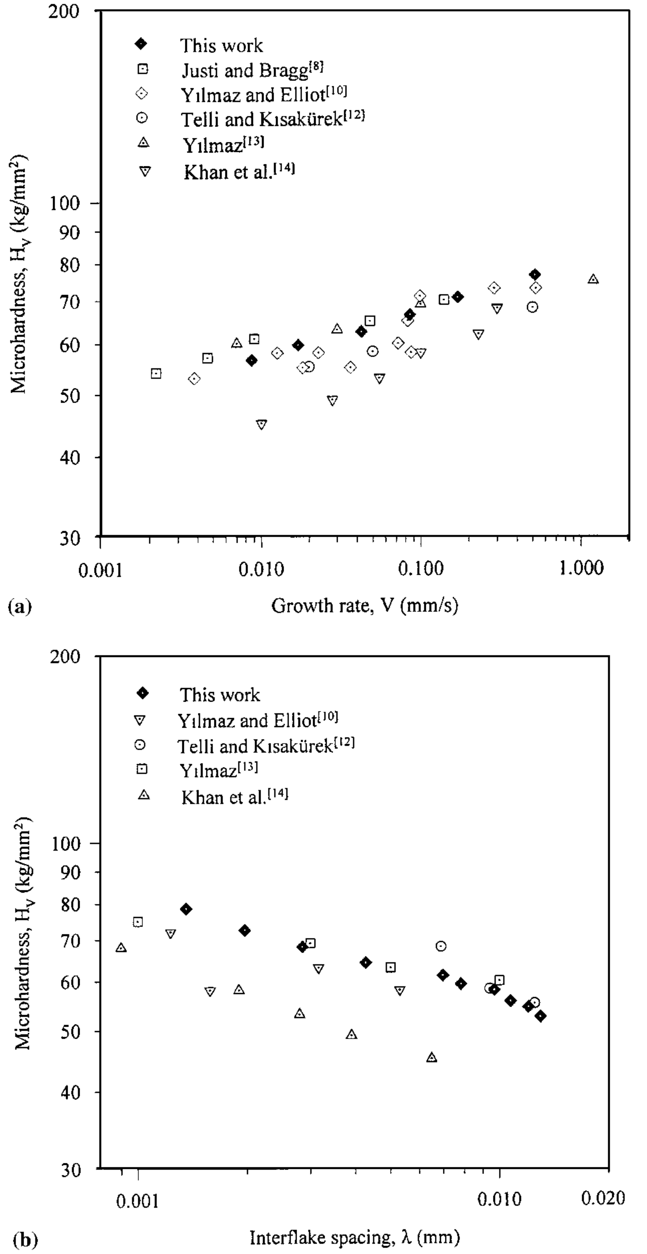

Fig. 5 Comparison of H_V values obtained with experimental data available in the literature for Al-Si eutectic alloys. **(a)** Variation of microhardness H_V, with growth rate, V; **(b)** variation of microhardness H_V, with lamellar spacing, λ.

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