

The Ag-Al (Silver-Aluminum) System

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Equilibrium Diagram

The Ag-Al equilibrium diagram has been studied extensively, and its general features are well established. There is little doubt that the equilibrium phases have been identified completely and correctly. However, some phase boundary location problems remain. The liquidus from 50.0 to 80.0 at.% Al, the solidus in general, the (Ag) solidus in particular, and the boundaries and structure of the μ phase need further study.

The equilibrium phases of the Ag-Al system are:

- the liquid, L
- the Ag-rich terminal solid solution, (Ag), with maximum solubility of about 20.4 at.% Al at about 450 °C
- the Al-rich terminal solid solution, (Al), with maximum solubility of about 23.5 at.% Ag at 567 °C
- the cph intermediate phase, δ , stable below about 727 °C, extending from about 23 at.% Al at 611 °C to 41.9 at.% Al at 567 °C
- the high-temperature bcc intermediate phase, β , stable above about 603 °C and extending from about 20.5 at.% Al at 779 °C to 29.8 at.% Al at 727 °C

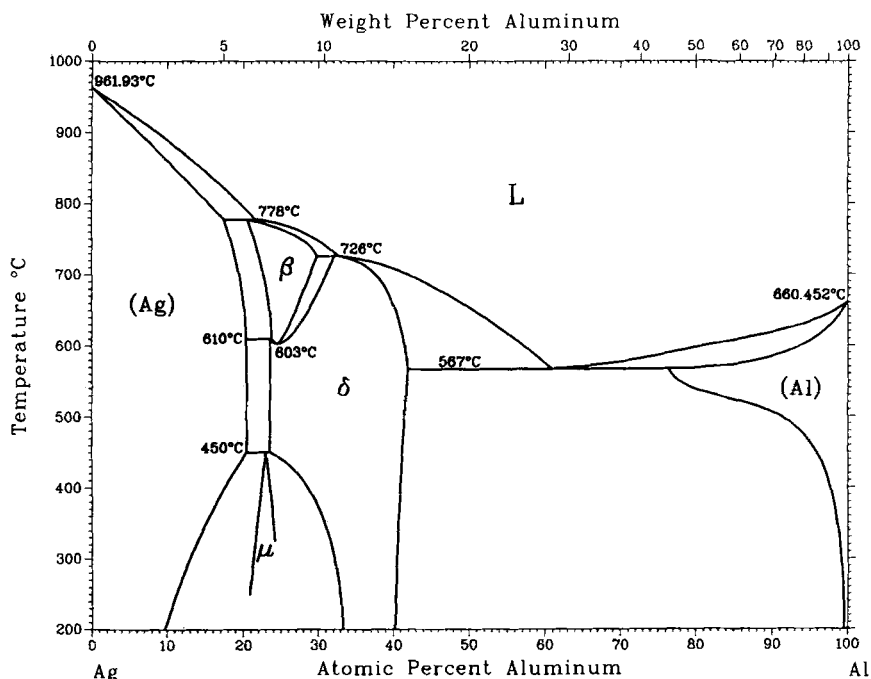
- the low-temperature intermediate phase, μ , reported to have complex cubic β Mn structure, stable below about 448 °C, with the single-phase field extending from about 21.2 to 24.3 at.% Al at 300 °C

The provisional diagram is shown in Fig. 1, and invariant reaction temperatures and compositions are listed in Table 1. The temperatures are averages of the values of [05Pet], [31Hoa], [40Hum], [41Fool], [49Ray], [50Owe], [70Mas], and [78Rob]. The compositions are extrapolations of phase boundaries to the invariant temperatures.

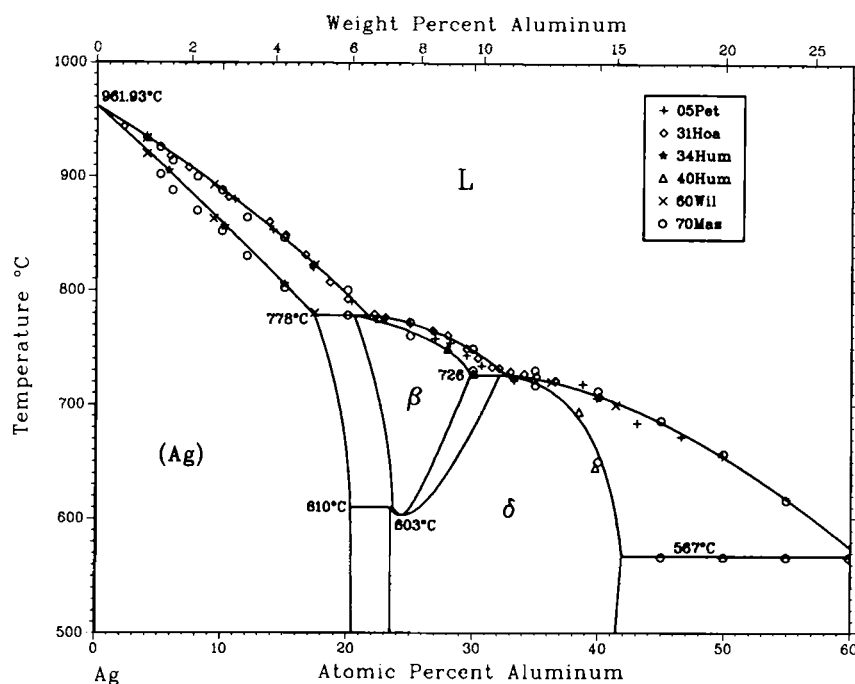
Liquidus. Many thermal analysis studies of the liquidus have been reported [05Pet, 28Han, 29Cre, 31Hoa, 33Tis, 37Tis, 39Tis, 40Che, 78Rob]. The liquidus was also determined by the electrochemical cell method [60Wil, 70Mas]. After thorough comparison, some of the studies were rejected on the basis of large scatter and/or inconsistency with the overall body of liquidus data. The accepted data [05Pet, 28Han, 31Hoa, 60Wil, 70Mas, 78Rob] are shown in Fig. 2 and 3. The data are sparse from 50 to 70 at.% Al, with consequent difficulty in ascertaining the $L \leftrightarrow \delta + (Al)$ eutectic composition.

Solidus. At least five attempts were made to determine the (Ag) solidus by thermal analysis [31Hoa, 50Pet] electrochemical cell [60Wil, 70Mas] and metallography

Fig. 1 Assessed Ag-Al Phase Diagram



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Fig. 2 Ag-Rich Liquidus and Solidus of (Ag), β and δ 

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Table 1 Special Points of the Assessed Ag-Al Phase Diagram

Reaction	Compositions of the respective phases, at.% Al			Temperature, °C	Reaction type
$L \leftrightarrow (Ag)$	0			961.93	Melting point
$(Ag) + L \leftrightarrow \beta$	17.4 ± 1.0	21.6 ± 0.5	20.5 ± 0.5	778 ± 2	Peritectic
$\beta + L \leftrightarrow \delta$	29.8 ± 1.0	32.7 ± 1.0	32.1 ± 1.0	726 ± 3	Peritectic
$(Ag) + \beta \leftrightarrow \delta$	20.3 ± 1.0	23.7 ± 0.5	23.5 ± 0.5	610 ± 3	Peritectoid
$\beta \leftrightarrow \delta$	24.4 ± 0.4			603 ± 4	Congruent
$L \leftrightarrow \delta + (Al)$	61.0 ± 2.0	41.9 ± 0.5	76.5 ± 0.5	567 ± 1	Eutectic
$(Ag) + \delta \leftrightarrow \mu$	20.4 ± 1.0	23.5 ± 0.7	22.9 ± 1.0	450 ± 3	Peritectoid
$L \leftrightarrow (Ag)$	100			660.452	Melting point

[34Hum]. The thermal analysis data were disregarded, because at lower temperatures and higher Al contents, they were in clear disagreement with the Al solubility limit established on samples subjected to long heat treatment. Slow equilibration of (Ag) with the melt is indicated. The (Ag) solidus data of [70Mas] were also suspect. They lay at lower temperatures than the data of [34Hum] and [60Wil] and displayed a curvature of opposite sign. The curvature of the [70Mas] data appeared to be incompatible with the thermodynamic properties of the liquid, (Ag), and (Al), in that it implied an implausible metastable extension of the fcc solidus. It should be noted, however, that the corresponding [70Mas] liquidus points tended to lie below the provisional liquidus; some small, consistently made compositional errors could therefore account for the apparent curvature.

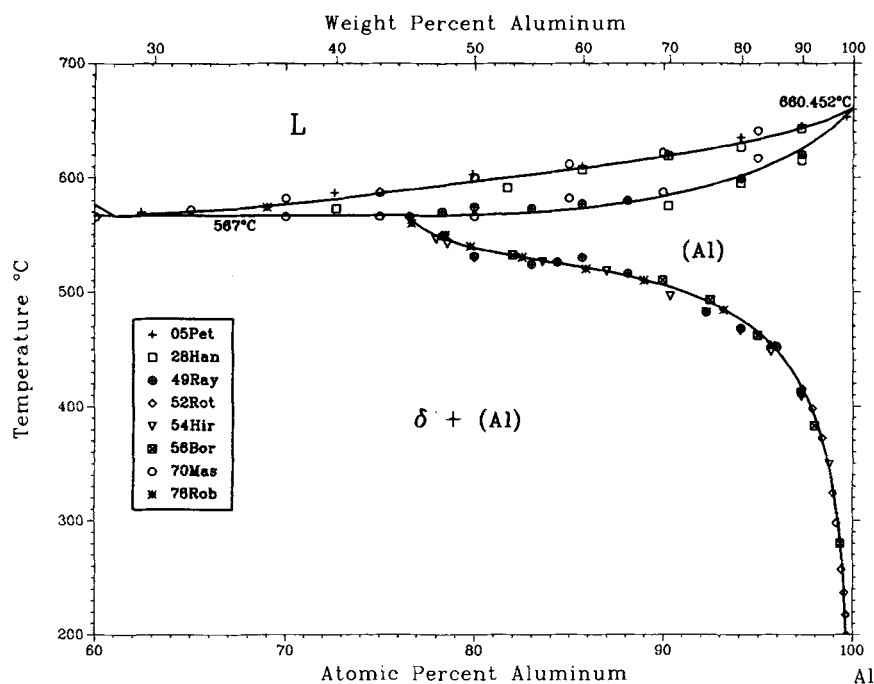
Three experimental β solidus points were obtained, two by [40Hum] by thermal analysis (heating) of samples equi-

librated in the single-phase β field, and one by [70Mas] with the electrochemical cell method. A fair curve, compatible with the terminating invariant temperature and compositions, can be drawn through these data.

The δ solidus was studied by [40Hum], using metallography and thermal analysis (heating) of samples equilibrated in the single-phase δ field. [70Mas] also studied this part of the solidus, by the electrochemical cell method, and their results were in excellent agreement with those of [40Hum].

The (Al) solidus was studied by thermal analysis (cooling [28Han], heating [37Tis]), metallography [49Ray], and electrochemical cell method [70Mas]. Each of these four sets of data showed noticeable scatter, but there were no inconsistencies among them. The solidus data are shown in Fig. 2 and 3.

Fig. 3 Liquidus, Solidus, and Solvus of (Al)



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(Ag) Solvus. The (Ag) solvus was investigated metallographically [31Hoa, 34Hum, 40Hum, 75Bar] and by the X-ray lattice parameter method [33Age, 41Foo, 50Owe]. With the exception of the [33Age] data, good agreement existed. All but the [33Age] data are shown in Fig. 4.

(Al) Solvus. There is little doubt that the (Al) solvus is inflected, because of the proximity of the incoherent (Al) miscibility gap (see below) to the solvus, as suggested by [49Ray]. Close scrutiny of the (Al) solvus data of [48Wak] and [49Ray] (both metallographic), [52Rot] (metallography and resistometry), [56Bor] (resistometry), [54Hir] (specific heat), and [78Rob] (microanalysis of heat treated samples), coupled with the X-ray diffraction studies and thermodynamic model potential analysis of [74Wil], makes this conclusion compelling. A few points in this body of data can be interpreted otherwise (as can the earlier measurements of [28Han] and [29Cre]), but great sensitivity to the presence of impurities is to be expected in this region of the diagram. In the freezing of such alloys, minor impurities can readily segregate to the melt, leaving the terminal liquid rich in impurities and resulting in a spurious freezing point. Anomalous points of [56Bor] at 70.0 and 75.0 at.% Al can be interpreted readily in this way. The data of [48Wak], [49Ray], [52Rot], [56Bor], [54Hir], and [78Rob] are shown in Fig. 5. Several (Al) solvus points were obtained by [70Mas] in their electrochemical cell studies. These data displayed an inflected solvus, but were in very poor temperature agreement with the other work. [70Mas] suggested that equilibrium was not attained in their measurements in this temperature/composition range.

β . Early workers [33Age, 36Obi, 39Tis] interpreted the reaction at 610 °C as a eutectoid ($\beta \leftrightarrow (\text{Ag}) + \delta$). However, high-temperature X-ray diffraction studies by [36Hof] indicated

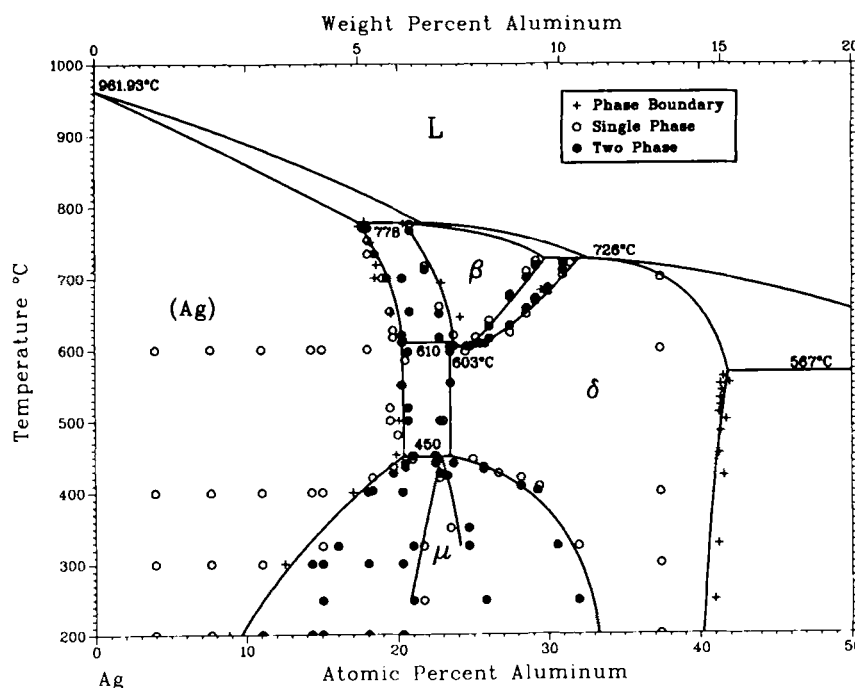
that the single-phase β field extends down to at least 605 °C at 25 at.% Al. Very careful, detailed microscopic and X-ray studies by [40Hum] established that the 610 °C reaction is peritectic ($(\text{Ag}) + \beta \leftrightarrow \delta$), and that the $\beta \rightarrow \delta$ transformation occurs below 604 °C and between 24.6 and 25.8 at.% Al. [40Hum] could not establish the nature of the $\beta \rightarrow \delta$ transformation. They suggested it is either congruent or a eutectoidal decomposition of β into δ of two compositions. The latter was thermodynamically implausible. The former was favored by thermal analysis studies [71Haw] of the massive $\beta \rightarrow \delta$ transformation.

The Ag-rich boundary was studied metallographically by [31Hoa] and [40Hum]. The Al-rich boundary was obtained metallographically by [40Hum] and electrochemically by [70Mas]. These data were in good agreement and are shown in Fig. 4.

The difficulty of retaining β in quenched massive samples is well known, but did not impede metallographic studies, because regions of transformed β are readily distinguishable.

δ . The Ag-rich δ boundary was determined metallographically [31Hoa, 40Hum, 75Bar], electrochemically [70Mas], and via the lattice parameter method [41Foo]. The Al-rich boundary was determined metallographically and by differential thermal analysis [40Hum], electrochemically [70Mas], and microprobe analysis [78Rob]. These data were in quite good agreement and are shown in Fig. 4.

μ . The boundaries of μ are based on the metallographic studies of [40Hum] and [75Bar], whose data are displayed in Fig. 4. On the basis of internal friction, dilatation, and resistometric measurements of 25 at.% Al, [71Yev] confirmed the peritectoidal formation of μ from (Ag) and δ and suggested that μ may order below 350 °C.

Fig. 4 Solid-State Boundaries of (Ag), β , μ , and δ 

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Metastable Phases

Aging of Al-rich Ag-Al alloys quenched from the fcc solid solution or from the liquid has been studied extensively. From this body of data, it is clear that two types of Guinier-Preston (GP) zones occur, GP-I forming above $\sim 170^\circ\text{C}$, and GP-II, below. A number of reversible property differences distinguish the two temperature regimes. Above 170°C in the GP-I region, diffuse X-ray scattering displays strong halos around the direct beam and Bragg peaks. Aging below 170°C produces asymmetric shells of scattering around superstructure positions [42Gui, 65Aue, 71Gra]. Samples aged below 170°C showed greater hardness [52Kos1] and lower electrical resistivity than those aged above 170°C , given zones of the same size [56Bor, 61Bor, 65Ave, 65Kos].

Low-angle X-ray scattering studies and electrical resistivity measurements [56Bor, 62Bau, 65Aue, 74Dau, 77Nau] indicated that GP-II zones have higher Ag content than GP-I zones. The latter data are plotted in Fig. 5. Moreover, on heating, GP-II zones transform endothermically into GP-I zones [52Kos2, 53Hir]. The reversible nature of the diffuse scattering and other properties led to the suggestion (e.g., [65Aue]) that the zones are ordered below 170°C . However, [71Gra] measured the diffuse X-ray scattering from a single crystal of 5.0 at.% Ag aged at 110°C on an absolute scale and were able to separate the data into components due to static and thermal displacements and to clustering. They found that the halving near superstructure positions was caused by static displacements, rather than ordering within the zones.

[71Gra] employed their clustering data in a computer simulation using 108000 atoms in an fcc lattice. The results

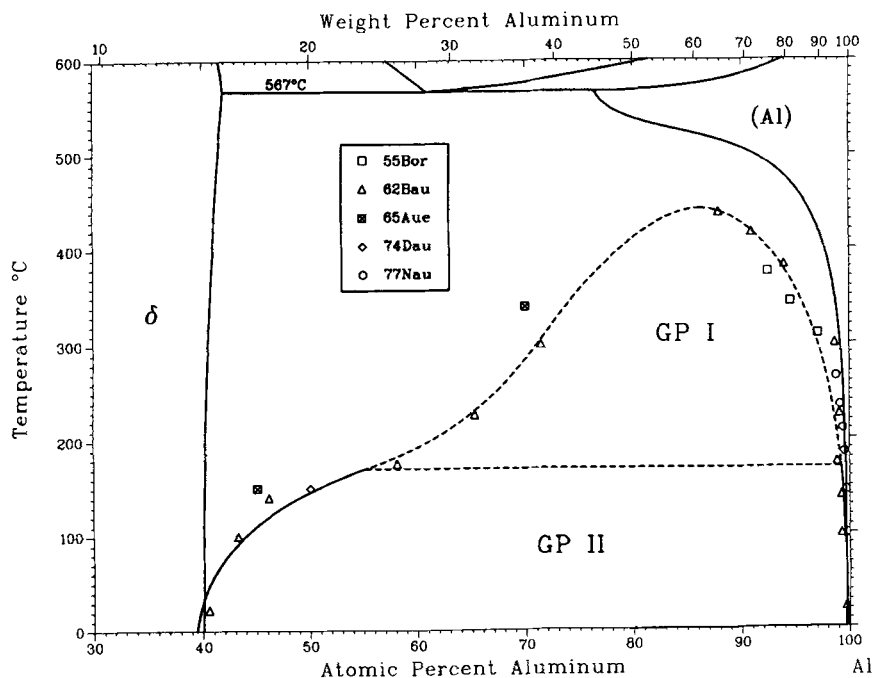
indicated that the zones are octahedral, bounded by $\{111\}$ planes and containing ~ 68 at.% Ag. This predicted Ag content at 110°C can be compared with the experimental value (Fig. 3) at 110°C [65Aue, 77Nau] of about 57.0 at.% Ag. Both $\{111\}$ facets and less prominent $\{100\}$ facets were observed by [84Ale] in a 5.1 at.% Ag sample aged at 160°C in the GP-II region. On further aging in the GP-I region (at 220, 285, 305, and 330°C), faceting persisted, but decreased markedly and (within the experimental scatter) smoothly with increasing temperature, giving no indication of a discontinuity between 160 and 220°C .

The nature of the driving force for the reversible GP-II \leftrightarrow GP-I transformation is not clear. No order-disorder transformation or abrupt change in zone shape or crystal habit occurs. Rather, below 170°C , zones richer in Ag, with resultant static lattice displacements, are favored energetically.

In addition to the GP zones, metastable cph δ' occurs in aging of Al-rich alloys. It appears at aging times longer than those required for the initiation of zone formation and grows in the form of Widmanstätten plates. δ' is partially coherent with the matrix, with principal stress in the basal plane that lies parallel to the $\{111\}$ fcc matrix planes [41Bar, 63Hrel]. Growth continues until coherence is lost, and transformation into equilibrium δ occurs. Observations of δ' growth established that heterogeneous nucleation occurs at stacking faults at dislocations in the matrix [61Fra, 61Nic, 63Hrel]. It was also suggested that δ' may form homogeneously from the zones [72Dob, 76Sch], but such arguments appeared to be based on models of the atomic structure within the zones that were incompatible with the results of [71Gra].

Electron diffraction and transmission electron microscopy studies of samples containing 21.0, 22.5, and 23.5 at.% Al

Fig. 5 Coherent Miscibility Gap in the $\delta + (Al)$ Phase Field



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Table 2 Ag-Al Crystal Structure Data

Phase	Homogeneity range, at.% Al	Pearson symbol	Space group	Strukturbericht designation	Prototype	Reference
(Ag)	0	<i>cF4</i>	<i>Fm$\bar{3}m$</i>	A1	Cu	[Pearson2]
β	20.5 to 29.8	<i>cI2</i>	<i>Im$\bar{3}m$</i>	A2	...	[Pearson2]
δ	22.9 to 41.9	<i>hP2</i>	<i>P6$\frac{3}{2}$/mmc</i>	A3	Mg	[Pearson2]
μ	~ 21 to 24	<i>cP20</i>	<i>P4$\frac{1}{2}$32</i>	A13	β Mn	[Pearson2]
(Al)	100	<i>cF4</i>	<i>Fm$\bar{3}m$</i>	A1	Cu	[Pearson2]

(a) At ~ 300 °C.

splat quenched from 1000 °C (just above the single-phase β field) were reported [72Gup, 73Gup]. Massive α_m , μ_m , and δ_m products, as well as three martensitic structures structurally compatible with the β phase, were observed, each occurring to some extent at all three compositions. Evidently, β is the first solid phase to form on quenching. No mention was made of retained β , nor were assessments of the relative amounts of massive and martensitic products reported.

These observations of massive α_m , μ_m , and δ_m in splat quenched samples were consistent with the results of studies on conventionally quenched samples [68Haw, 69Ari, 71Haw]. The [72Gup] martensite with hexagonal structure (the 1H stacking variant) is presumably identical to the hexagonal martensite observed conventional quenching from β reported by [69Ari] and [71Haw], who were in good agreement on the martensite transformation temperature:

$$M_s = 2867 - 11868xAl \text{ } ^\circ\text{C}$$

where $0.244 \leq xAl \leq 0.250$, and the cooling rate is about 10^{-5} °C/s.

[72Gup] reported all three martensites formed in splat quenching to be disordered. The shape memory effect was reported by [75Kub], who quenched their samples of 24.1 at.% Al from 700 °C in the β field in ice water to retain β , then requenched to temperatures ranging from -36 to -89 °C. Martensite of two different morphologies was observed in the second phase of quenching, but no structural identifications were made. [75Kub] noted that ordered martensite is necessary for the shape memory effect to occur.

Crystal Structures and Lattice Parameters

Ag-Al crystal structure and lattice parameter data are listed in Tables 2 and 3. The bcc structure of β was established by high-temperature X-ray studies [36Hof, 36Obi]. The phase can be retained by sufficiently rapid quenching [68Haw,

Table 3 Ag-Al Lattice Parameter Data

Phase	Composition, at.% Al	Lattice parameters, nm		Comment	Reference	
		a	c			
(Ag)	0	0.40862	...	At RT	[Pearson2]	
		0.40861	...	At RT	[41Foo]	
	4.00	0.40811	...	At RT	[41Foo]	
	10.76	0.40727	...	At RT	[41Foo]	
	11.22	0.40720	...	At RT	[41Foo]	
	14.24	0.40680	...	At RT	[41Foo]	
	15.75	0.40661	...	At RT	[41Foo]	
	17.35	0.40643	...	At RT	[41Foo]	
	17.45	0.40642	...	At RT	[41Foo]	
	18.28	0.40638	...	At RT	[41Foo]	
	β	25.00	0.324	...	At 700 °C	[36Hof]
		25.74	0.3302	...	At RT	[36Obi]
	μ	25.7(a)	0.629	...	At RT	[36Obi]
	δ	26.3	0.2871	0.4665	At RT	[59Mas]
		28.6	0.2874	0.4677	At RT	[59Mas]
30.3		0.2875	0.4633	At RT	[59Mas]	
32.8		0.2878	0.4618	At RT	[59Mas]	
34.05		0.2879	0.4612	At RT	[59Mas]	
34.87		0.2880	0.4606	At RT	[59Mas]	
35.85		0.2881	0.4601	At RT	[59Mas]	
36.70		0.2882	0.4591	At RT	[59Mas]	
39.00		0.2884	0.4585	At RT	[59Mas]	
(Al)		85.4	0.41057	...	At 525 °C	[62Sim]
		87.2	0.41051	...	At 525 °C	[62Sim]
		90.0	0.41039	...	At 525 °C	[62Sim]
	92.4	0.41030	...	At 525 °C	[62Sim]	
	95.0	0.41024	...	At 525 °C	[62Sim]	
	97.5	0.41014	...	At 525 °C	[62Sim]	
	98.9	0.41000	...	At 525 °C	[62Sim]	
	100.0	0.40982	...	At 525 °C	[62Sim]	
		0.40496	...	At RT	[Pearson2]	

(a) Alloy in two-phase μ + δ region, mostly μ.

69Ari, 71Haw]. Lattice parameters obtained at high temperatures are listed in Table 3.

The lattice parameters of cph δ obtained by [59Mas] on samples quenched from 550 °C are listed in Table 3. In a sample containing 33.2 at.% Al annealed at 150 °C for 10 days, [66Neu] observed strong short-range order within the basal plane and weak long-range order in the c direction. They were unable to present a satisfactory structural model.

Early workers [28Wes, 36Hof, 36Obi, 37Kat] reported complex cubic βMn structure for μ. The most recent study by [49Rob] indicated cubic structure of lower symmetry. The lattice parameters of (Al) listed in Table 3 were obtained at 525 °C by [62Sim]. Many attempts have been made to obtain (Al) lattice parameters from samples quenched from either the solid or the liquid [48Axo, 50Gul, 52Ell, 55Hil, 62Hel, 73Rob]. Little significance can be attached to these results, because even the most rapidly quenched samples show some degree of spinodal decomposition [73Rob].

Thermodynamics

[69Bel] measured the Ag and Al partial Gibbs energies of the liquid at 1600 °C from 6.1 to 90.2 at.% Al by the vapor pres-

sure technique. Electrochemical cell measurements of the Al partial Gibbs energy of the liquid were made by [60Wil] from 3.9 to 97.3 at.% Al between 700 and 900 °C and by [70Mas] from 0.5 to 95.0 at.% Al between the liquidus and 1050 °C. When the Al liquid partial Gibbs energy data of [60Wil] and [70Mas] are extrapolated to 1600 °C, they are found to be in very good agreement with the Al partial Gibbs energy data of [69Bel].

The enthalpy of mixing of the liquid was studied by reaction calorimetry by [30Kaw] from 14.6 to 80.1 at.% Al at 1050 °C and by [68Ita] from 10.0 to 90.0 at.% Al at 970 °C. Drop-calorimetry measurements of the liquid heat of mixing were done from 30 to 90 at.% Al at 800 °C by [69Bej] (unpublished work tabulated by [70Cas]). These three sets of liquid enthalpy of mixing data were in poor mutual agreement, with the [60Bej] data in very strong disagreement with the others for Al contents less than 50 at.%. None of the three was in good agreement with the enthalpy of mixing calculated from the electrochemical cell Al partial Gibbs energy measurements of [60Wil] and [70Mas]. Determination of the Al partial Gibbs energy of mixing in (Ag) were carried out from 5.2 to 18.4 at.% Al between 347 and 547 °C [56Hil], from 3.9 to 17.4 at.% Al between 700 and 980 °C [60Wil], and from 5 to 20 at.% Al between 350 °C and the liquidus [70Mas]. These

three sets of data were in reasonable agreement, but the question of equilibrium attainment at low temperatures is problematical.

[56Hil] determined the Al partial Gibbs energy of mixing for (Al) from 80.4 to 95.4 at.% Al at 547 °C. [70Mas] measured the Al partial Gibbs energy of mixing in δ from 30.0 to 40.0 at.% Al between 350 °C and the liquidus.

The heat of solution of Ag in liquid Al was measured calorimetrically by [67Mat] to be 18630 J/mol of atoms. [69Wit] measured the heat of formation of (Ag) from 7.1 to 17.6 at.% Al, of δ from 25.0 to 39.0 at.% Ag, and of (Al) from 96.2 to 98.2 at.% Al at 470 °C, from the heats of solution of Ag, Al, and Ag-Al alloys in liquid Zn.

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*Indicates key paper.

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