Aluminium – Boron – Carbon

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Literature Data

Due to the presence of carbon contaminant in aluminium borides the data on the constitution of the Al-B-C ternary system and those of the binary Al-B system have to be reviewed carefully, which was done by [1994Dus] to strictly differentiate between true aluminium borides and aluminium boron carbides.
High hardness in combination with high neutron absorption, high wear resistance and impact resistance have triggered an early interest in high-strength and low-weight Al-B_4C composite materials or cermets, either in bulk form with a metal binder or by reinforcing an Al-base matrix with boron carbide particles or with boron carbide-coated fibres. Understanding the phase equilibria proved of major importance in the processing of Al-B_4C composites particular in finding processing criteria at temperatures high enough to promote wetting and low enough to control reactions and design microstructures.
Despite much effort was spent on the synthesis and crystallographic characterization of the various ternary aluminum boron carbide compounds [1964Mat, 1965Eco, 1965Mat, 1966Gie, 1966Lip, 1969Per, 1969Wil, 1970Nei, 1977Mat, 1987Sar, 1980Ino, 1990Oka, 1992Via, 1994Kud, 1995Osc, 1996Hil1, 1996Hil2, 1997Mey], information on the equilibrium phase relations in the Al-B-C ternary system is scarce [1993Bau, 1997Via, 1998Rog]. These informations comprise early calculations of the phase equilibria disregarding the boron-rich compounds or rather assuming the phases, $AlB_{40}C_4$ and $Al_{2.1}B_{51}C_8$, to be part of the solid solution range of "B_4C" [1982Doe, 1993Kau]. Some confusion in the early experimental work on aluminum borides arose from the fact that due to contamination either from high carbon level boron starting material or from the use of graphite crucibles and substrates aluminum boron carbides were produced rather than binary aluminum borides. This is particularly true for "AlB_{10}" [1963Wil] - shown to be "$AlB_{24}C_4$" or more precisely $Al_{2.1}B_{51}C_8$ [1964Mat, 1967Wil, 1969Wil, 1969Per, 1990Oka] - and (βAlB_{12}) [1960Koh], later shown to be $Al_3B_{48}C_2$ [1965Mat]. According to structural and DTA investigations [1996Hil1], $Al_3B_{48}C_2$ exists in a tetragonal high temperature modification, which on cooling below 650°C transforms into a body-centered orthorhombic low temperature phase with a unique structure type. The mixture of two orthorhombic phases with coherent boundary and commensurable lattice parameters (modifications A and B), as claimed by [1965Mat, 1986Pes], thus simply explains by multiple twinning on cooling [1996Hil1].
An experimental study of the isothermal section at 1400°C by [1993Bau] confirmed the existence of four ternary compounds $Al_{2.1}B_{51}C_8$ [1964Mat, 1967Wil, 1969Wil, 1969Per], $AlB_{40}C_4$ [1966Gie, 1966Lip, 1970Nei], $Al_3B_{48}C_2$ [1996Hil1] and Al_3BC_3 [1996Hil2]. The latter compound was first mentioned as "$Al_4B_{1-3}C_4$" [1964Mat] and later labelled as "$Al_8B_4C_7$" [1980Ino] from a cursory investigation of its crystal symmetry with X-ray single crystal photographs, although no details of the crystal structure were derived. The relation to a wurtzite structure was discussed [1995Osc]. A structure determination is due to [1996Hil2]. A fifth compound, Al_3BC [1992Via, 1993Gon, 1997Mey, 2002Zhe], (earlier "Al_4BC" [1987Sar, 1989Hal, 1990Pyz]) was reported to exist below ~1000°C [1992Via], however, was shown in the isothermal section at 1000°C [1997Via]. From a detailed analysis (XPD, LOM, SEM, EPMA) of the Al-rich corner [1997Via] on about 30 specimens prepared from cold pressed and sintered powder compacts in the temperature region from 627 to 1000°C, an isothermal section at 1000°C and a tentative liquidus projection was derived assisted by a series of isothermal diffusion experiments by heating together in an alumina boat an Al-B rod and an Al-C rod.
Phase equilibria at 900°C in the Al-C rich part of the ternary Al-B-C system were established [2002Zhe] from XPD of about 45 ternary and binary alloys. Equilibrium conditions were not reached for boron-rich samples. An attempt to obtain equilibrated samples from mixtures B_4C+AlB_2, B_4C+Al and B_4C+Al_4C_3 were also unsuccessful. Al_3BC and Al_4C_3 phases form very easily and are observed in all samples even after short time sintering in contrast to Al_3BC_3, which forms very slowly at 900°C. On the other hand Al_3BC_3 was always observed in arc melted samples containing 40-60 at.% Al and 10-30 at.% B.

Experimental techniques for preparation concerned (a) melting of B_4C in excess of Al for the synthesis of $AlB_{40}C_4$ (at 1550°C, [1970Nei]), (b) melting of boron with excess of Al in a graphite crucible for synthesis of $Al_3B_{48}C_2$ (at 1400°C, [1964Mat]) (c) vapor deposition at 1400 to 1600°C for single crystals of $Al_3B_{48}C_2$ [1967Bli] (d) hot pressing of B_4C+Al in graphite dies for synthesis of $Al_{2.1}B_{51}C_8$ (at 1800°C [1966Gie, 1966Lip]), (e) infiltration of B_4C by liquid Al at 1100°C and anneal at 1000°C to obtain Al_3BC [1987Sar], (f) reaction sintering of Al+B+C powder compacts on alumina boats in sealed silica capsules 627 to 1000°C for the synthesis of Al_3BC and phase relations at 1000°C [1992Via, 1997Via] or at 1400°C for 10 h for the production of the single crystals of $Al_3B_{48}C_2$ [1994Kud] (g) melting of an Al_8BC mixture in alumina under argon for 160 h at 850°C and subsequent cooling at 150K/h to RT for production of black-bluish single crystals of Al_3BC [1997Mey] (h) melting of an $Al_{40}B_2C_3$ mixture in alumina under argon at 1500°C and subsequent cooling at 10K/h to 600°C for production of single crystals of Al_3BC_3 in the form of yellow, transparent platelets [1996Hil2] and (i) Al-flux solvent method for a general production of single crystals (see i.e. [1986Kis, 1990Oka, 1996Hil1]). Samples used for the isothermal section at 1400°C were prepared from cold compacted powder mixtures of AlB_2, B_4C, B and/or C, which were reaction-sintered under Ar in closed Knudsen-type graphite reactors at 1600°C for 1h prior to 48 h heat treatment at 1400°C [1993Bau]. Phase relations at 900°C were studied [2002Zhe] on elemental powder compacts sintered in alumina crucibles (binary Al-B alloys) or in closed graphite crucibles (ternary alloys). The specimens were sealed in evacuated quartz ampoules and were slowly heated for 10°/h to 720°C (slightly above the melting point of aluminium) and kept at this temperature for 48h. After temperature was increased to 900°C at a rate of 20°/h, the tablets were sintered at this temperature for 1 week. Repeated repowderisation (under protective cyclohexane) and sintering at 900°C were necessary to reach equilibrium conditions. Several studies dealt with the kinetics of wetting of B_4C surfaces by liquid aluminum; detailed discussions can be found in the articles by [1979Pan] and [1989Hal]. Hot-pressing of $B_{4.3}C$+Al powders at 1820°C, 45 MPa under Ar (5 to 20 mass% Al) revealed the formation of the ternary B_4C- related Al-boron carbides (solution of Al in B_4C, and τ_2) although the products were all thought to belong to the B_4C-based solid solution [2000Liu]. With increasing Al-content (>5 mass% Al) the Al_3BC_3 phase evolved [2000Liu].
Thermodynamic calculations of the Al-B-C system have been attempted by [1982Doe, 1993Wen, 1993Kau], however, are not fully consistent with experimental observations. Reviews on the constitution and on the crystal structures of the Al-B-C system have been presented by [1977Mat, 1990Luk, 1998Rog].

Binary Systems

The binary systems B-C and Al-C are consistent with the critical assessments of [1996Kas] and [2003Per], respectively. In spite of numerous data available from literature on the constitution of the Al-B phase diagram, contradictory results exist for the formation of aluminium diboride (Table 1). Fig. 1a shows the various versions for the Al-rich part of the Al-B phase diagram. It should be noted, that recent experiments [2002Zhe] confirmed the formation of AlB_2 at 900°C, in contrast to data of [1997Via] suggesting peritectic formation at 892 ± 5°C. In the present assessment we accept the temperature of 956 ± 5°C for the invariant reaction $L+AlB_{12} \rightleftharpoons AlB_2$ as determined by [2000Hal]. The adopted Al-B phase diagram (Figs. 1b, 1c) is based on the assessment of [1994Dus]. The composition of the peritectic liquid at 0.55 at.% B has been confirmed by a recent thermodynamic assessment of [2001Fje]. AlB_2 is still taken as a stoichiometric compound in spite of the suggestions of [1964Mat, 1999Bur, 2002Bur] for Al-deficiency in terms of $Al_{0.9}B_2$.
Although the assessment of [1994Dus] concluded a peritectic formation of AlB_{12}, $L+(B) \rightleftharpoons AlB_{12}$ at 2050°C, the thermodynamic calculation of [1993Wen] is based on congruently melting AlB_{12} (T_M = 2150°C).

Solid Phases

The crystallographic information on all the binary and ternary phases pertinent to the Al-B-C system is listed in Table 2. Some controversy exists in the crystallographic characterization of the modifications reported for $Al_3B_{48}C_2$. A single crystal study [1995Hil, 1996Hil1, 2000Mey] on an untwinned specimen revealed a tetragonal high temperature form (closely related to the structure of I-tetragonal boron), which on cooling undergoes a symmetry reduction resulting in microscopically twinned products that hitherto

were indexed on the basis of two orthorhombic modifications, labeled A and B by [1965Mat]. The transformation was earlier proposed to be at ca. 850°C [1960Koh, 1965Mat], whereas new results from DTA recorded 650°C [1996Hil1]. The transition seems to be rather fast, as the low temperature modification is present in samples furnace-cooled from 1400°C to room temperature [1993Bau].
A second point of controversy concerns the phases $AlB_{40}C_4$ and $Al_{2.1}B_{51}C_8$ for which detailed crystallographic descriptions are available, however, $AlB_{40}C_4$ actually being isotypic with binary B_4C, hitherto is not thoroughly established as a ternary phase independent from binary B_4C. As the two structurally closely related phases $AlB_{40}C_4$ and $Al_{2.1}B_{51}C_8$ generally are found together, a high and low temperature transition between them may be inferred [1993Bau]. Without further details the maximum solid solubility of Al in boroncarbide ("$B_{13}C_2$", at 1950°C) was reported to be 1 mass% Al (equivalent to 2 at.% Al in B_4C) [1978Ekb].
Experiments to establish a possible homogeneous range for Al_3BC_3 (earlier: "$Al_8B_4C_7$" [1980Ino], or "$Al_4B_{1-3}C_4$" [1964Mat]), were carried out at 1830°C by [1980Ino] resulting in a rather stoichiometric composition without variation of the lattice parameters. These findings were confirmed by [1993Bau, 1996Hil2]. Details of the crystal structure with linear C-B-C chains are given by [1996Hil2]. Lattice parameters of Al_3BC_3 were measured at room temperature up to 7.5 GPa using a multi-anvil synchrotron system with B_4C anvils; for a high temperature pressure experiment the sample was placed in a graphite ampoule [2000Sol]. Al_3BC_3 is free of structural transitions up to 1523°C within the pressure range 2.5 to 5.3 GPa [2000Sol]. A further ternary compound τ_5 was observed after infiltration by liquid Al at 1170°C with post heat treatment for 100 h at 800 to 1000°C [1987Sar]. The hexagonal lattice was established by TEM; the approximate composition "$Al_{\sim 4}BC$" resulted from EELS-data [1987Sar]. This phase has been also confirmed by [1989Hal, 1990Pyz]. From a detailed investigation of this Al-rich boroncarbide by X-ray powder diffraction, LOM and EPMA, [1992Via] suggested a formula of Al_3BC rather than "Al_4BC" and attributed a hexagonal cell; additional weak lines in the X-ray intensity pattern of Al_3BC prompted a larger unit cell $a = a_0/\sqrt{3}$ [1993Gon]. Although the authors of [1997Mey] recognized the larger cell, the crystal structure of Al_3BC was solved for the high symmetry subcell from single crystals isolated from a sample directly reacted from the elements - however, from EPMA a composition $Al_{2.5}BC$ was derived (see also Table 2). Al_6B-octahedra and trigonal Al_5C-bipyramids are the characteristic structural elements [1997Mey].
The various data on the compositional ranges of the τ_4 and τ_5 phases are summarized in Fig. 2. Half filled circles correspond to the accepted stoichiometries Al_3BC and Al_3BC_3.
From the significant change of the unit cell volume of Al_4C_3 comparing binary and ternary alloys, a solubility of boron is suggested [1996Bid, 2002Zhe, 2000Mey]. Solubility of boron in Al_4C_3 was established to be 3.4 at.% at 900°C [2002Zhe] and an interesting behavior of lattice parameters was observed. In spite of the increase of the "*a*" parameter and of the cell volume with boron content, the "*c*" parameter decreases. That may be explained by a preferential distribution of boron and carbon atoms among different crystallographic sites. A significant solubility of boron in Al_4C_3 was also reported by [2000Mey] to be about 9.3 at.%, however, no details on the relevant temperature were given. Furthermore these authors claim for $Al_4C_{3-x}B_x$ lattice parameters increasing with boron content. Lattice parameters of Al_4C_3 for samples located in three phase regions (Al)+Al_4C_3+Al_3BC, Al_4C_3+Al_3BC_3+Al_3BC and Al_4C_3+Al_3BC_3+(C) are very close, assuming that these three phase regions meet at the Al_4C_3 phase at a maximal boron solubility of $Al_4(C_{0.92}B_{0.08})_3$.
Insignificant solubility of carbon in AlB_2 is reported by [2002Zhe] comparing lattice parameters in ternary and binary samples; AlB_2 with 0.5 at.% C, heat treated at 900°C, already contains the Al_3BC phase.

Isothermal Sections

Phase equilibria for the 1400°C isothermal section are summarized in Fig. 3, revealing the existence of four ternary compounds τ_1 to τ_4. A small field of liquid phase exists at 1400°C which is in equilibrium with $Al_3B_{48}C_2$, $Al_{2.1}B_{51}C_8$ and with Al_3BC_3 [1993Bau]. Boron-poor equilibria agree with an earlier work by [1980Ino] who reported on the two-phase equilibria Al_4C_3+Al_3BC_3 ($Al_7B_4C_8$), Al_3BC_3 ($Al_7B_4C_8$)+B_4C and Al_3BC_3 ($Al_7B_4C_8$)+C. In Fig. 3 two-phase equilibria are shown to exist between the binary solid

solution "B_4C" and $AlB_{40}C$ and $Al_{2.1}B_{51}C_8$. At 1400°C all ternary compounds seem to exist at their stoichiometric compositions [1993Bau], whilst [1965Eco] claimed a homogeneity range for τ_1 at 1800°C from "$AlB_{48}C_8$" to $Al_3B_{48}C_8$. Binary AlB_{12} was never seen in combination with $Al_{2.1}B_{51}C_8$ nor with $AlB_{40}C_4$ [1993Bau].
The isothermal section at 1000°C, Fig. 4, was constructed on the basis of data from [1997Via]. Due to low interaction kinetics in the boron- and carbon-rich part of the system at 1000°C, equilibria in this portions of the diagram are preliminary. Moreover, ternary compounds τ_1 and τ_2 were not included in the 1000°C section by [1997Via], τ_4 was listed as "$Al_8B_4C_7$", and no solubility of boron in Al_4C_3 was considered. For consistency with the present knowledge on the Al-B-C system, the ternary compounds τ_1 and τ_2 were introduced in Fig. 4 and the composition of τ_4 was changed to Al_3BC_3. The solubility of boron in Al_4C_3 at 1000°C was estimated to be about 4 at.%, extrapolating from data of [2002Zhe] at 900°C. Figure 5 represents the isothermal section at 900°C [2002Zhe] confirming the equilibrium AlB_2+Al_3BC, whereas [1997Via] claimed this equilibrium to be only stable below 868 ± 4°C. Similar to 1000°C the equilibria at 900°C involving τ_1 and τ_2 are not well established due to low reactivity of the components.

Invariant Equilibria, Liquidus Surface

A tentative liquidus surface for the aluminum rich portion of the diagram (Fig. 6) was proposed by [1997Via], presenting equilibria involving the τ_5 phase. The invariant equilibrium U_5 ($L+Al_3B_{48}C_2 \rightleftharpoons AlB_2+Al_3BC$) was reported at 868 ± 4°C by [1997Via], but this temperature can not be accepted in respect to the observed isothermal equilibrium AlB_2+Al_3BC at 900°C [2002Zhe] suggesting such transformation above 900°C. Comparison of the reaction scheme and the isothermal section at 1000°C (Fig. 4) with the isothermal section at 1400°C (Fig. 3) suggests a rather complicate picture of the phase transformations in this regions mainly due to decomposition of τ_5.
Based on an earlier thermodynamic calculation by [1982Doe], a reaction scheme was derived [1990Luk], which gives a tentative information of the solidification behavior in the Al-B-C ternary. The temperatures of the invariant equilibria were estimated and the ternary compounds τ_1 to τ_3 were assumed to be part of the solid solution arising from binary B_4C; τ_5 was not included. A more recent thermodynamic modelling of the Al-B-C phase diagram by [1993Wen] as part of the multi-component Al-B-C-N-Si-Ti system treated the ternary compounds τ_1, τ_2, τ_3 as independent phases, however, the peritectoid formation of τ_4 (Al_3BC_3) is in strict contradiction to the experimentally confirmed two-phase equilibrium τ_4+τ_5 (Al_3BC_3+Al_3BC) [1997Via, 2002Zhe] as well as to the observed existence of τ_4+τ_5 in as cast alloys [2002Zhe], thereby strongly indicating direct formation of Al_3BC_3 from the liquid.
A closed ternary miscibility gap in the Al-rich liquid is suggested from thermodynamic calculations by [1993Kau], however, hitherto without experimental confirmation [2002Zhe].
Figure 7 presents a reaction scheme for the major parts of the Al-B-C phase diagram. The reaction scheme is essentially based (i) on the tentative liquidus projection for the Al-rich part as suggested by [1997Via], (ii) on the experiments of [2002Zhe] concerning the solidification of the phases τ_4, τ_5 and (iii) on the thermodynamic calculation of [1993Wen] for the B-rich part, however, accepting peritectic formation of AlB_{12}.

Thermodynamics

Enthalpies of formation and heat capacity measurements from a Calvet type automatic microcalorimeter in the temperature range 310-1200 K were reported by [1987Kis] and are listed as follows:
$Al_3B_{48}C_2$: $H^0(T) - H^0(298) = 0.7945 \cdot 10^{-3} T^2 + 0.5182T - 225.1374$ (in $J \cdot g^{-1}$) and
$C_p(T) = 0.1589 \cdot 10^{-2} T + 0.5182$ ($J \cdot g^{-1} K^{-1}$)
$Al_{2.1}B_{51}C_8$: $H^0(T) - H^0(298) = 0.7226 \cdot 10^{-3} T^2 + 0.5411T - 225.5702$ (in $J \cdot g^{-1}$ for $AlB_{24}C_4$) and
$C_p(T) = 0.1589 \cdot 10^{-2} T + 0.5182$ ($J \cdot g^{-1} \cdot K^{-1}$ for $AlB_{24}C_4$).
Thermodynamic calculations of the Al-B-C system are due to [1982Doe, 1993Wen, 1993Kau], however, are not fully consistent with experimental observations. For detailed discussion, see section Invariant Equilibria.

Notes on Materials Properties and Applications

Mechanical properties of Al-B_4C cermets and boron/carbon fiber-aluminium composites have been investigated by various groups [1972Bak, 1973Her, 1975Mun, 1984Via, 1985Che, 1985Hal, 1985Kov, 1985Pyz, 1985Sar, 1986Che, 1986Dub,1990Ram,1996Pyz, 2002Ars]; the effect of reaction on the tensile behavior of infiltrated composites was reported by [2002Kou2] and size dependent strengthening in particle reinforced Al by [2002Kou1]; reaction products were studied by [2001Lee]. An increase of surface hardness of about 25 to 40 % can be achieved by impulse laser radiation on B_4C/Al cermets [1988Kov].

Wetting of B_4C by Al has been studied by many research teams with rather contradicting results, until the temperature and time dependent occurrence of chemical reactions/compound formation was analyzed in detail (for discussion see i.e. [1979Kis, 1979Pan, 1989Hal, 2000Kha]). The kinetics of wetting by liquid aluminium of flat, sintered boron carbide specimens with residual porosity less than 3 % were investigated by [1979Pan]. The speed of spreading of liquid aluminium at 1100° to 1200°C was measured to be 0.1-0.8 $mm \cdot s^{-1}$, in accordance with $r^2 = f(t)$, where r equals the radius of the contact circle. The angle of contact was first ~92°, however, in 3 to 5 min decreased to 28°. The slow spreading was determined by the formation of new aluminum boron carbide phases in the contact zone with a microhardness of ca. 13 GPa. The driving force $\Delta\sigma = \sigma (\cos \Theta_0 - \cos\Theta)$ (σ = surface tension of the melt, Θ_0 = contact angle of the melt, Θ=contact angle at time (*t*)) decreased sharply becoming zero in 4 to 5 min [1979Pan]. The contact angle of molten Al on B_4C as a function of processing time for various isotherms at $5 \cdot 10^{-3}$ to 10^{-4} Pa was also given by [1989Hal] based on sessile drops cooled to room temperature.

Mechanical properties, electrical and thermal conductivity as well as their temperature dependencies were reported on the Knoop and Vickers microhardness for Al-flux grown (temperature region 1750 to 800°C) ''amber'' single crystals $Al_3B_{48}C_2$ and for ''black'' crystals (αAlB_{12}, γAlB_{12} and $AlB_{2.1}B_{51}C_8$) [1986Kis]. These studies were also performed on hot pressed specimens of various compositions $x(AlB_{12})+(1-x)B_4C$ and $Al_3B_{48}C_3$ in the temperature range 24 to 827°C [1991Kha1, 1991Kha3]. For Al_3BC_3 (”$Al_8B_4C_7$”), $Al_3B_{48}C_3$, $Al_{2.1}B_{51}C_8$ [1991Kha2] also examined these properties as a function of porosity and quantity of Fe-impurity. These data are summarized in Table 3 including information on flux-grown crystals $Al_3B_{48}C_2$ and $Al_{2.1}B_{51}C_8$ [1990Oka]. Both types of crystals were said to be p-type semiconductors [1986Kis].

In a ring test the strength of a powder compact of $B_{13}C_2$ +1 mass% Al, sintered at 1950°C, was found to be 0.50(7) GNm^{-2} [1978Ekb]. [1991Kha2] reported on the kinetics of thermal densification of hot pressed powders of B_4C, AlB_{12}, $Al_3B_{48}C_2$ and Al_3BC_3. Kinetics of dissolution in HCl, HNO_3 and HCl-HNO_3 was studied by [1998Kha] as well as the resistance of Al-boron carbides to alkali and hydrogen peroxide. [1989Hal] studied the densification kinetics of Al+B_4C cermets in the range from 800 to 1400°C in pressureless sintering as well as after applying hot isostatic pressure. The kinetic of metal depletion in post heated dense cermets B_4C/Al at temperatures between 600°C and 1000°C was investigated by [1990Pyz]. Chemical stability against various boiling acids, oxidation resistance, IR and EPR spectra of Al-borides and Al-boron carbides ($Al_3B_{48}C_2$, $Al_{2.1}B_{51}C_8$) was studied by [1991Pri]. The spectra were taken at 77K and 300K and for different crystal orientations relative to the magnetic field. Absorption edge and IR-active phonons in $Al_3B_{48}C_2$ were reported by [1987Hau, 2000Wer] and IR spectra of boron carbide containing up to 1.5 at.% Al were determined between of 8 to 500 mm^{-1} wave numbers and for temperatures between 70 to 450 K [1997Sch]. These data seem to suggest the incorporation of Al-atoms into binary boron-carbide in form of pairs substituting the B-B-C or C-B-C chains [1997Sch]. Characteristic IR absorption bands for finely dispersed powders of Al-borides and Al-boron carbides were listed by [1998Kha].

The Seebeck-coefficients were reported to linearly increase from 260 μVK^{-1} for binary ”B_4C” to 450 μVK^{-1} for 1.4 at.% Al dissolved, revealing p type behavior [1997Sch]. Seebeck-coefficients, thermal and electric conductivities were further reported by [2000Liu] for $B_{4.3}C$-based samples containing 0.5, 10, 15, 20 mass% Al, highlighting the Z-value at RT of $1.04 \cdot 10^{-6}$ K^{-1} for the 5 mass% Al sample. IR and Raman spectroscopy on Al_3BC_3 (at RT) confirm the linear $(CBC)^{5-}$ unit as an isoelectronic CO_2-analogon [1996Hil2, 2000Mey].

On heating in air, Al_3BC_3 (earlier reported as $Al_8B_4C_7$), $Al_3B_{48}C_3$ and $Al_{2.1}B_{51}C_8$ show low oxidation at 500°C (increase of mass ~4 mgh^{-1}); intensive oxidation, with a mass increase of ~40 mgh^{-1}) starts at

1280°C for $Al_{2.1}B_{51}C_8$ and at 1370°C for $Al_3B_{48}C_2$ [1991Pri, 1989Kha, 1991Kha4]. Oxidation in air of single crystals $Al_{2.1}B_{51}C_8$ and $Al_3B_{48}C_2$ started at about 760°C and 710°C, respectively [1990Oka]. The reaction products were $9Al_2O_3{\cdot}2B_2O_3$ for $Al_{2.1}B_{51}C_8$ crystals and B_2O_3 for $AlB_{40}C_4$ specimens [1994Kud]. Whereas Al_3BC_3 was said to be unstable in acids [1991Kha4], more detailed experiments [1996Hil2] proved stability at room temperature against bases and dilute acids, except for HNO_3 and HF. Al_3BC_3 was furthermore said to be stable in air up to 600°C [1991Kha4,1996Hil2]. Al_3BC is quickly attacked by dilute HCl [1997Mey].

Thermophysical properties of sintered bodies of Al_3BC_3 have been derived by [2000Wan]. These are linear thermal expansion in the range of 25 to 1200°C, specific heat and thermal diffusivity via laser flash technique, Youngs modulus of 136.6 GPa, Vickers hardness of 12.1 GPa at a load of 196 N and thermogravimetric recording of growth of an oxidized layer on heating in air up to 1500°C.

Fitting a Birch-Murnaghan equation of state to the pressure dependency of the lattice parameters of Al_3BC_3 up to 7.5 GPa, the isothermal bulk modulus was B_0=153 ± 6 GPa (dB_0/dp=19 ± 4) [2000Sol]. Despite high bulk modulus the Vickers hardness of single crystals is as low as 20.7 GPa at a load of 25g and 18.2 GPa at a load of 50g [2000Sol].

Al_3BC was successfully prepared by self propagating high temperature synthesis induced by mechanical activation of Al-B-C powder mixtures in air; mixtures low in boron ($AlB_{0.1}C$) resulted in Al_3BC_3 under violent emission of heat [1999Tsu]. In contrast to that [2000Sav] was unable to prepare ternary aluminoborocarbides from mechanochemical synthesis. Elastic bulk and shear moduli for Al_3BC (earlier reported as Al_4BC) were measured by [1995Pyz] and estimated by [1999Tor].

Miscellaneous

A series of patents covers the techniques to produce dense B_4C/Al cermets by infiltration of the metal matrix into the porous ceramic body without wetting reactions [1976Lan, 1986Hal, 1987Pyz, 1990Pyz, 1991Pyz, 1995Pyz, 1996Pyz, 1997Du, 2000Pyz, 2001Lee]; subsequent heat treatment results in materials with designed chemistry and microstructures, flexure strength, hardness and fracture toughness. Fine microstructures were obtained via ultrarapid microwave heating [1995Rug]. B_4C/Al cermets have been considered as an improved structural neutron absorber [1977Ros, 1978Boi, 1978Sur, 1986Ros, 1987Lev, 1992Bei] and for applications as friction materials for automotive brake applications [1999Cha]. Oxidation protective B_4C-coatings on C-fibers in Al-matrix were reported by [1996RMi] and [1996Vin] produced C-fibres-Al composites by a squeeze casting technique. Explosive consolidation to produce Al/B_4C composites was studied by [1995Bon, 1997Yue]. Shock recovery experiments were performed on a 65 vol% B_4C-Al cermet as a function of shock pressure [1989Blu].

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Table 1: Literature Data on Experimental Temperatures of Invariant Equilibrium $L+AlB_{12} \rightleftharpoons AlB_2$

Technique	Heating Rate	*T* [°C]	References
Stability observation	-	1000 -1500	[1936Hof]
Synthesis observation	-	980	[1967Ser]
DTA	4°C/min	920	[1967Ato]
Stability observation	-	1350-1500	[1972Sir]
DTA	5°C/min	1030±5	[1993Ips]
Synthesis observation	-	892±5	[1997Via]
DSC and Stability observation	0°C/min*	956±5	[2000Hal]
DSC	10°C/min	914±55	[2001Fje]

* DSC measurements were performed with heating rate of 5, 15 and 40°/min., and extrapolated to 0°C/min.

Table 2: Crystallographic Data of Solid Phases

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
(Al) < 660.452	*cF*4 $Fm\bar{3}m$ Cu	*a* = 404.96	[Mas2]
(βB) < 2092	*hR*333 $R\bar{3}m$ βB	*a* = 1093.30 *c* = 2382.52 *a* = 1092.2 *c* = 2381.1 *a* = 1096.5 *c* = 2386.8 *a* = 1097.4 *c* = 2387.7	[Mas2, 1993Wer] at 1.1 at.% C [1993Wer] linear d*a*/d*x*, d*c*/d*x* at AlB_{31} [V-C2] from sample $Al_4B_{95}C_1$, quenched from 1400°C, contains $Al_3B_{48}C_2$ and αAlB_{12} [1993Bau]
(C) < 3827 (B.P.)	*hP*4 $P6_3/mmc$ C-graphite	*a* = 246.12 *c* = 670.90 *a* = 246.023 *c* = 671.163 *a* = 246.75 *c* = 669.78	[Mas2] [1967Low] at 2.35 at.% B_{max} (2350°C) linear da/d*x*, dc/d*x*, [1967Low]

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
B_4C	*hR*45	*a* = 565.1	9 to 20 at.% C [1990Ase]
< 2450	$R\bar{3}m$	*c* = 1219.6	
	$B_{13}C_2$	*a* = 560.7	
		c = 1209.5	
		a = 560.3	from sample containing τ_2, τ_4, quenched
		c = 1209.8	from 1400°C [1993Bau]
$B_{25}C$	*tP*52	*a* = 872.2	[V-C2]
	$P\bar{4}2m$	*c* = 508.0	also $B_{51}C_1$, $B_{49}C_3$; all metastable?
	$B_{25}C$		
Al_2B_3	*hR**	*a* = 1840	at 60 at.% B [1992Var]
≤ 525	Al_2B_3 (?)	*c* = 896	metastable?
AlB_2	*hP*3	*a* = 300.6	[1994Dus],
≤ 956±5	*P*6/*mmm*	*b* = 325.2	temperature from [2000Hal]
	AlB_2	*a* = 300.67 ± 0.01	[2002Zhe]
		b = 325.36 ± 0.02	
		a = 300.63 ± 0.01	[2002Zhe] in 33.3Al-66.2B-0.5C,
		b = 325.46 ± 0.01	in equilibrium with τ_5 at 900°C
		a = 300.43 ± 0.03	[1999Bur] for $Al_{0.9}B_2$
		b = 325.19 ± 0.06	
αAlB_{12}	*tP*216	*a* = 1015.8	[1994Dus]
≤ 2050	$P4_12_12$	*c* = 1427.0	$\rho_{exp.}$ = 2.65 Mgm^{-3}
	αAlB_{12}	*a* = 1018	[1991Pri]
		c = 1434.3	
		a = 1016.3	from sample $Al_2B_{92}C_2$, quenched from
		c = 1425.6	1400°C, contains $Al_3B_{48}C_2$ [1993Bau]
		a = 1015.5	from sample $Al_4B_{95}C_1$, quenched from
		c = 1426.0	1400°C, contains $Al_3B_{48}C_2$ and AlB_{31}
			[1993Bau]
		a = 1014.93 ± 0.07	[2002Zhe]
		c = 1425.0 ± 0.5	
γAlB_{12}	*oP*384	*a* = 1014.4	[1983Hig, 1994Dus, 2000Hig]
	$P2_12_12_1$	*b* = 1657.3	metastable phase or ternary product
	γAlB_{12}	*c* = 1751.0	stabilized by small amounts of impurity
			metals present in Al-flux grown material
		a = 1019.5	$\rho_{exp.}$ = 2.56 Mgm^{-3}
		b = 1666	[1991Pri]
		c = 1769	
Al_4C_3	*hR*21	*a* = 333.8	[2003Per, V-C2]
< 2156	$R\bar{3}m$	*c* = 2511.7	
	Al_4C_3	*a* = 334.21 ± 0.01	[2002Zhe]
		c = 2503.2 ± 0.5	
		a = 335.78 ± 0.02	[2002Zhe] in 57.1Al-4.3B-38.6C
		c = 2499.6 ± 0.5	$Al_4(C_{0.9}B_{0.1})_3$, in equilibrium with τ_5 at 900°C

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
* τ_1, $Al_{2.1}B_{51}C_8$ (eventually low temperature phase of τ_2)	*oC*88 *Cmcm* $Al_2B_{51}C_8$		earlier labeled "AlB_{10}" [1967Wil] or $AlB_{24}C_4$ [1964Mat,1969Wil,1970Wil]]
		a = 569.0 *b* = 888.1 *c* = 910.0	[1969Per] $\rho_{exp.}$ = 2.54 Mgm^{-3}
		a = 568.7 *b* = 887.7 *c* = 909.8	from sample containing τ_2 and τ_3, quenched from 1400°C [1993Bau]
		a = 569.0 *b* = 888.1 *c* = 910.0	from sample containing τ_4, quenched from 1400°C [1993Bau]
		a = 569.3 *b* = 884.7 *c* = 909.3	from sample $Al_4B_{92}C_4$ quenched from 1400°C, contains $Al_3B_{48}C_2$ (tetragonal), $Al_3B_{48}C_2$ (A) and $AlB_{40}C_4$ [1993Bau]
		a = 567.6 *b* = 891.4 *c* = 909.5	[1991Pri]
		a = 569.2 *b* = 889.2 *c* = 911.2	[1990Oka] $\rho_{exp.}$ = 2.54 Mgm^{-3} single crystals from Al-flux
* τ_2, $AlB_{40}C_4$ (eventually high temperature phase of τ_1)	*hR*45 $R\bar{3}m$ B_4C-deriv.	*a* = 564.2 *c* = 1236.7	[1970Nei] $\rho_{exp.}$ = 2.52 Mgm^{-3}
		a = 565.37 *c* = 1231.4	from sample containing τ_1, τ_3, quenched from 1400°C [1993Bau]
		a = 564.8 *c* = 1239.9	from sample containing τ_4 and B_4C, quenched from 1400°C [1993Bau]
		a = 565.6 *c* = 1238.9	from sample $Al_4B_{92}C_4$ quenched from 1400°C, contains also $Al_3B_{48}C_2$ (tetrag.), $Al_3B_{48}C_2$ and $Al_{2.1}B_{51}C_8$ [1993Bau]
		a = 563 *c* = 1129	[1966Gie] for composition "$Al_2B_{48}C_8$"
		a = 565 *c* = 1239	[1966Lip] for composition "$Al_4B_{48}C_8$"

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
* τ_3, $Al_3B_{48}C_2$ (r)	*oI*212	a_0 = 1240.7	[1996Hil1], only one low temperature
< 650	*Imma*	b_0 = 1262.3	modification!
	$Al_3B_{48}C_2$	c_0 = 1014.4	
		a = 1234	[1965Mat], two modifications,
		b = 1263	microscopically twinned;
		c = 508	modification A, $c=c_0/2$
		a = 1232.5	from a sample $Al_6B_{92}C_2$ cooled from
		b = 1261.4	1400°C contains "AlB_{12}" [1993Bau]
		c = 1016.2	
		a = 1233.72	from sample $Al_4B_{95}C_1$ cooled from
		b = 1262.41	1400°C contains also "AlB_{12}", AlB_{31}
		c = 1016.06	[1993Bau]
		a = 1232.5	[1991Pri]
		b = 1264.7	
		c = 1016.2	
		a = 1230.2	[1994Kud]
		b = 1262.1	
		c = 1016.1	
		a = 1229.1	from sample $Al_4B_{92}C_4$ cooled from
		b = 1262.2	1400°C, [1993Bau] contains
		c = 1015.88	$Al_{2.1}B_{51}C_8$, $AlB_{40}C_4$ and tetragonal $Al_3B_{48}C_2$
		a = 1233.62	[1993Bau] from sample $Al_4B_{95}C_1$,
		b = 1262.40	cooled from 1400°C, see above.
		c = 1015.94	
		a = 1239.0 ± 0.3	[2000Wer]
		b = 1263.7 ± 0.3	
		c = 1013.6 ± 0.4	
		a = 1237.7	[1990Oka]
		b = 1262.7	single crystals from Al-flux
		c = 507.9 to	modification A ; $c = c_0/2$
		a = 1236.3	
		b = 1261.6	
		c = 510.2	
		a = 616.6	[1990Oka]
		b = 1263.5	single crystals from Al-flux
		c = 1065.6	$\rho_{exp.}$ = 2.59(2) Mgm^{-3}
		a = 618.1	modification B, $a = a_0/2$
		b = 1262.2	
		c = 1016.1	
		a = 617	[1965Mat]
		b = 1263	modification B
		c = 1016	$a = a_0/2$
		a = 616.4	[1991Pri]
		b = 1262.1	$a = a_0/2$
		c = 1016.4	

Phase/ Temperature Range [°C]	Pearson Symbol/ Space Group/ Prototype	Lattice Parameters [pm]	Comments/References
* τ_3, $Al_3B_{48}C_2$ (h)	*tP*52	*a* = 885	[1996Hil1] high temperature
> 650	$P4_2/nnm$	*c* = 508	modification
	$B_{25}C$ -deriv.	*a* = 882	[1965Mat]
		c = 509	
		a = 881.9	from sample $Al_4B_{92}C_4$ cooled from
		c = 508.25	1400°C, contains also $Al_{2.1}B_{51}C_8$, $AlB_{40}C_4$ and orthorhombic $Al_3B_{48}C_2$ [1993Bau]
* τ_4, Al_3BC_3	*hP*42	*a* = 589.97	[1996Hil2] ρ = 2.66 Mgm^{-3}
< 1835	$P\bar{3}c1$ [a]	*c* = 1589.0	temperature from [1980Ino]
	Mg_3BN_3	*a* = 590.6	[1980Ino], labelled as $Al_8B_4C_7$
		c = 1590.1	
		a = 590.7	from sample containing τ_1,
		c = 1591.3	quenched from 1400°C [1993Bau]
		a = 590.5	from sample containing τ_2 and B_4C,
		c = 1590.5	quenched from 1400°C [1993Bau]
		a = 340.1 ± 0.3	[2000Sol], subcell with $a = a_0/\sqrt{3}$
		c = 1584 ± 0.2	pressure dependence of the lattice parameters is given up to 7.5GPa
		a = 590.22 ± 0.3	[2002Zhe]
		c = 1589.4 ± 0.1	
*τ_5, Al_3BC	*hP*20	*a* = 605.0	[1993Gon, 1997Via]
< 1100	$P\bar{3}c1$	*c* = 1154.0	
	($P6_3/mmc$	*a* = 603.45	[1997Mey] from single crystals,
	for subcell)	*c* = 1152.02	"$Al_{2.5}BC$" from EPMA
	Al_3BC	*a* = 6041.9 ± 0.2	[2002Zhe]
		c = 1154.0 ± 0.3	
		a = 349.1	[1992Via], subcell with $a = a_0/\sqrt{3}$
		c = 1154.1	
		a = 352.0	[1987Sar], earlier "Al_4BC"
		c = 582.0	subcell with $a = a_0/\sqrt{3}$, $c = c_0/2$

[a] $P6_3/mmc$ for subcell with $a = a_0/\sqrt{3}$, $c = c_0$

Table 3: Microhardness, Fracture Toughness, Electrical Conductivity, Activation Energies for Electrical Conductivity and Thermal Conductivity for Various Aluminium Borides and Aluminium Boron Carbides.

Compound	Crystal Face of Indent	Microhardness [GPa] at Various Loads and Temperatures				Fracture Toughness K_{1c} [MPa·m$^{1/2}$]	ρ_{293K} [Ωm]	Activation Energy ΔE [eV] 100K to 400K $R=R_0\exp(-\Delta E/2kT)$	Thermal Conductivity [Wm^{-1}K^{-1}]	References
		Knoop		Vickers						
$Al_3B_{48}C_2$	(111)	26.5(5)	(2N 293K)							[1986Kis]
		23.1	(5N, 293K)							[1986Kis]
	(100)					5.3	10^4-10^6	1	19.6 (310K)	[1986Kis]
		27.1(5)	(2N, 293K)	33.6(1.6)	(2N, 293K)	4(1)	$2.6\cdot10^3$-10^{-6}	0.6 - 1.2		[1991Pri]
				25.7 to						[1990Oka,
				30.5	(1N, 293K)		$2.6\cdot10^{-6}$			1994Kud]
	(111)	37.6(2.0)	(0.5N, 293K)							[1986Dub]
		31.7(8)	(1N, 293K)							[1986Dub]
		23.7(6)	(4.9N, 293K)							[1986Dub]
$Al_{2.1}B_{51}C_8$	(100)	22.6	(2N, 293K)				10^{-3} - 1	0.1	38.7 (310K)	[1986Kis]
		26	(5N, 293K)						60 (600K)	[1986Kis]
		6	(5N, 1200K)							[1986Kis]
		24.2(7)	(2N, 293K)			2.7(2)	$2.02\cdot10^5$	0.08 - 0.18		[1991Pri]
		25.0-26.9	(1N, 293K)							[1990Oka]
Al_3BC_3				20.7	(0.25N, 293K)					[2000Sol]
				18.2	(0.50N, 293K)					
αAlB_{12}	(101)	19.6(5)	(2N, 293K)			1.5(3)	$5.92\cdot10^2$	0.18 - 0.36		[1991Pri]
γAlB_{12}	(001)	29.6(1.0)	(0.5N, 293K)	34.4(2.7)	(0.5N, 293K)					[1986Dub]
		25.8(7)	(1N, 293K)	31.0(1.5)	(1N, 293K)					[1986Dub]
		22.8(8)	(2N, 293K)	27.3(1.2)	(2N, 293K)	1.8(2)	$3.85\cdot10^5$	0.22		[1991Pri]
		21.6(1.1)	(4.9N, 293K)	23.8(9)	(4.9N, 293K)					[1986Dub]

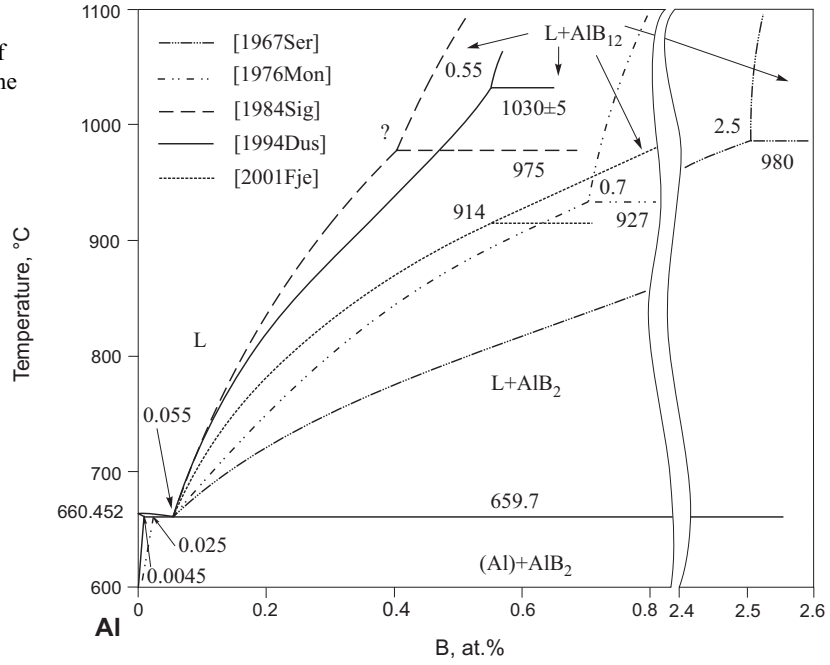

Fig. 1a: Al-B-C. Various versions of the Al-rich part of the Al-B diagram

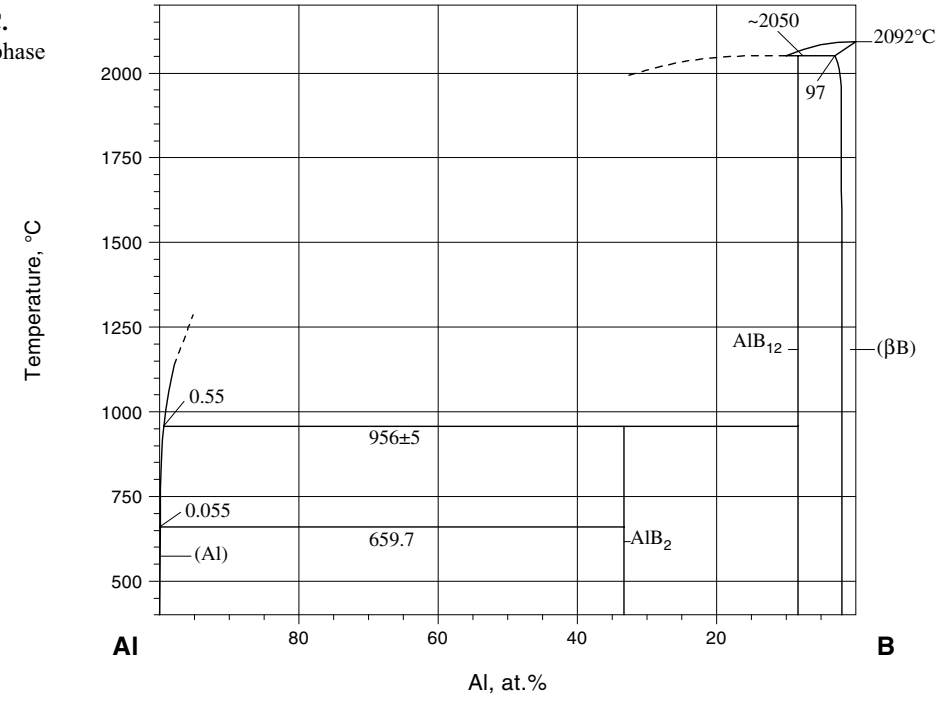

Fig. 1b: A-B-C. Accepted Al-B phase diagram

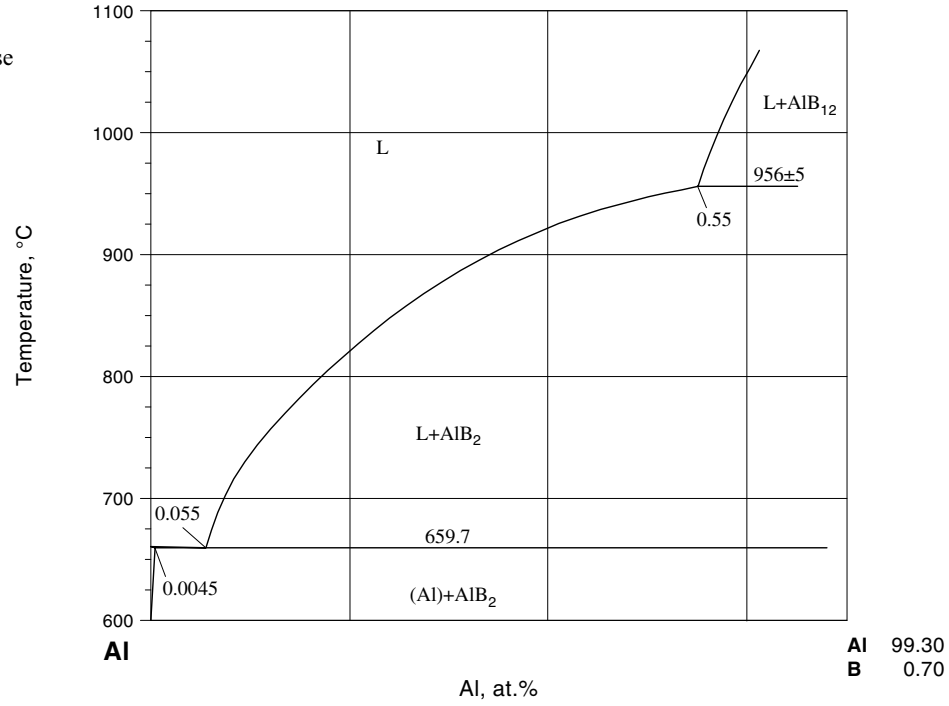

Fig. 1c: Al-B-C. Accepted Al-B phase diagram, enlarged Al-rich region

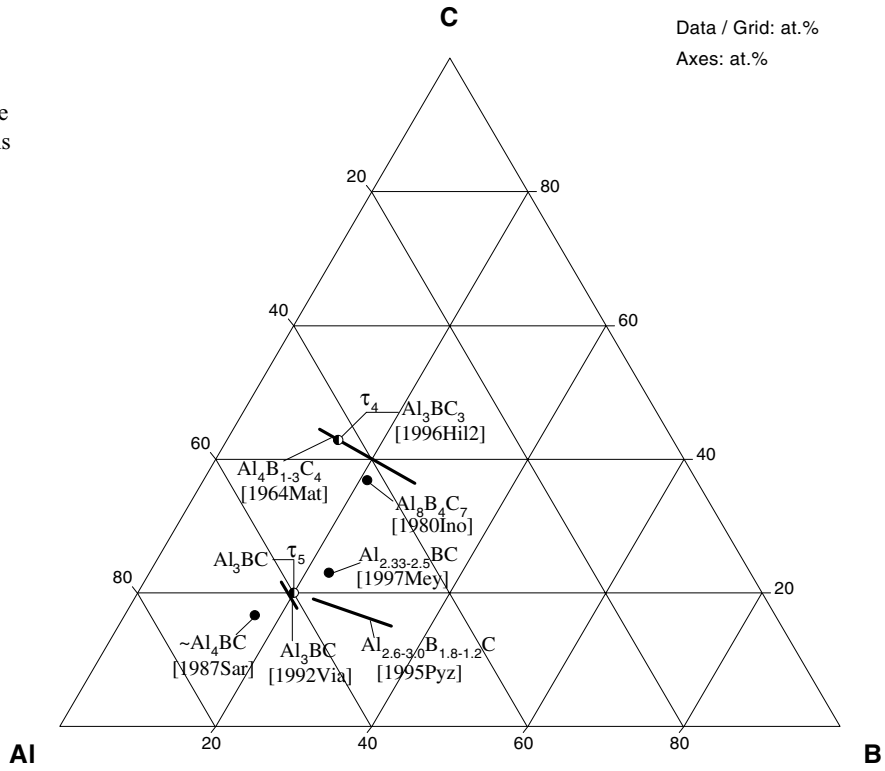

Fig. 2: Al-B-C. Superposition of literature data on the homogeneity regions of τ_4 and τ_5 phases. Half-filled circles correspond to the accepted compositions

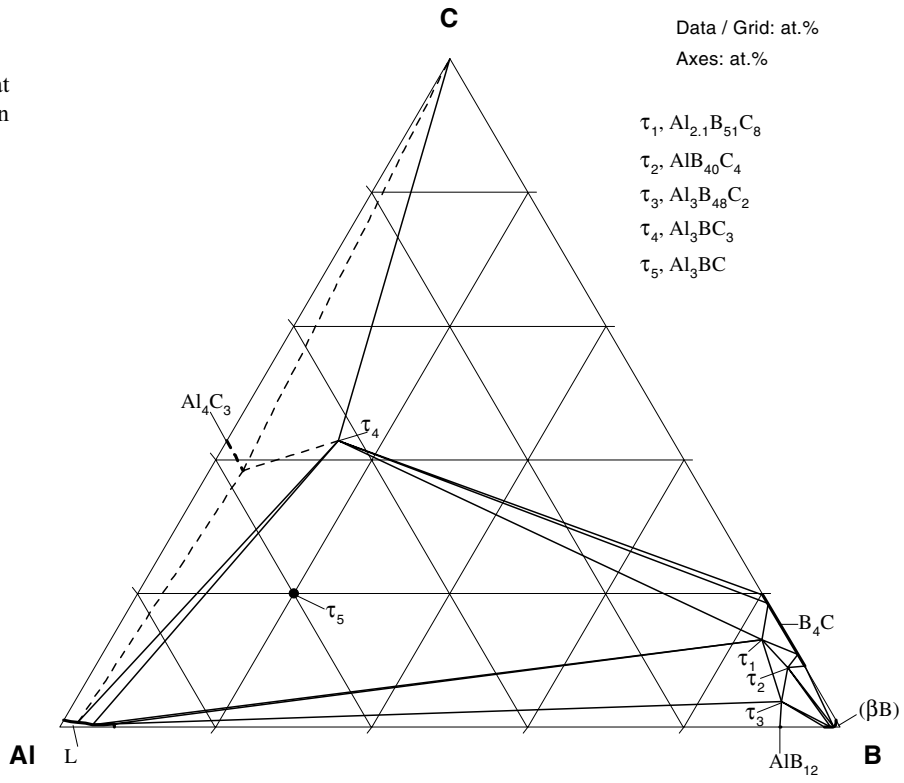

Fig. 3: Al-B-C. Isothermal section at 1400°C; the position of Al_3BC is indicated by a full circle

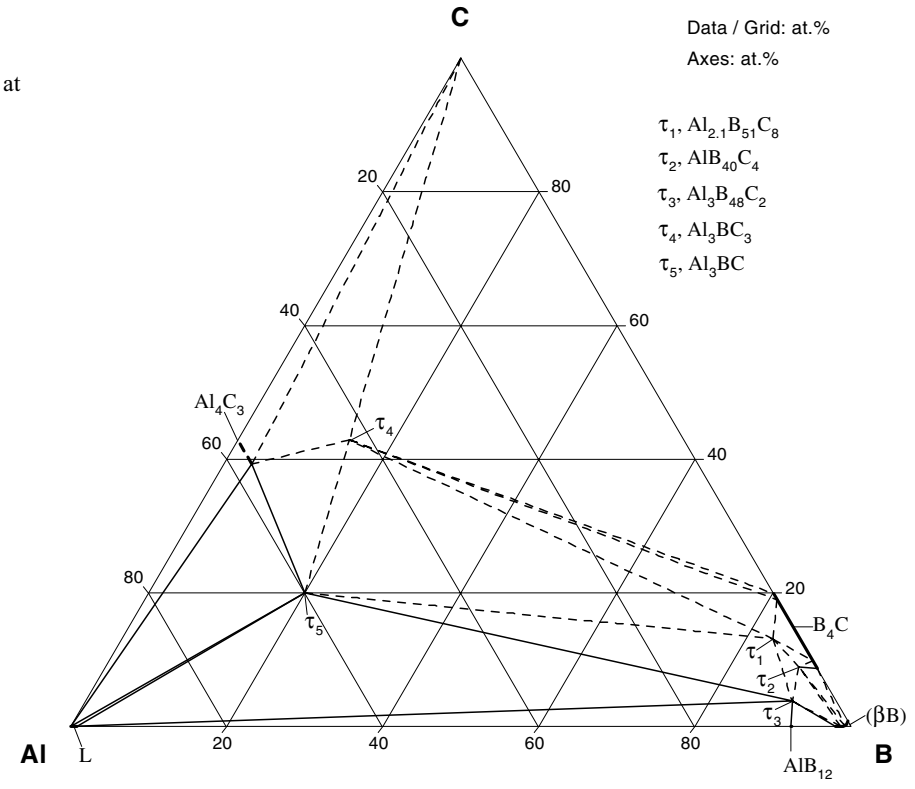

Fig. 4: Al-B-C. Isothermal section at 1000°C

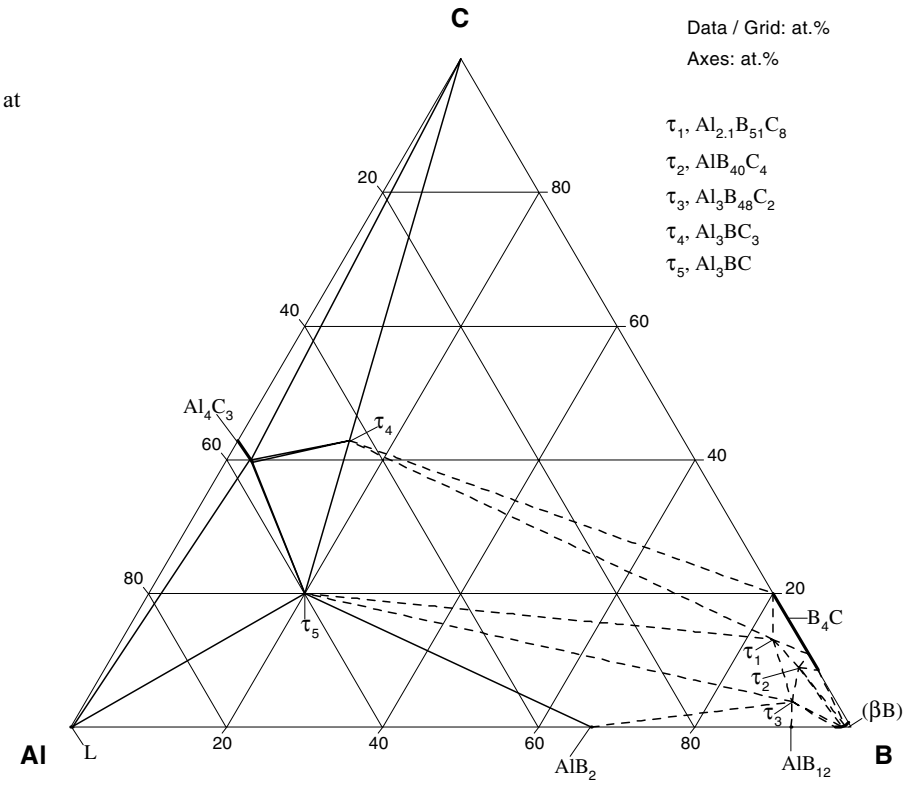

Fig. 5: Al-B-C.
Isothermal section at 900°C

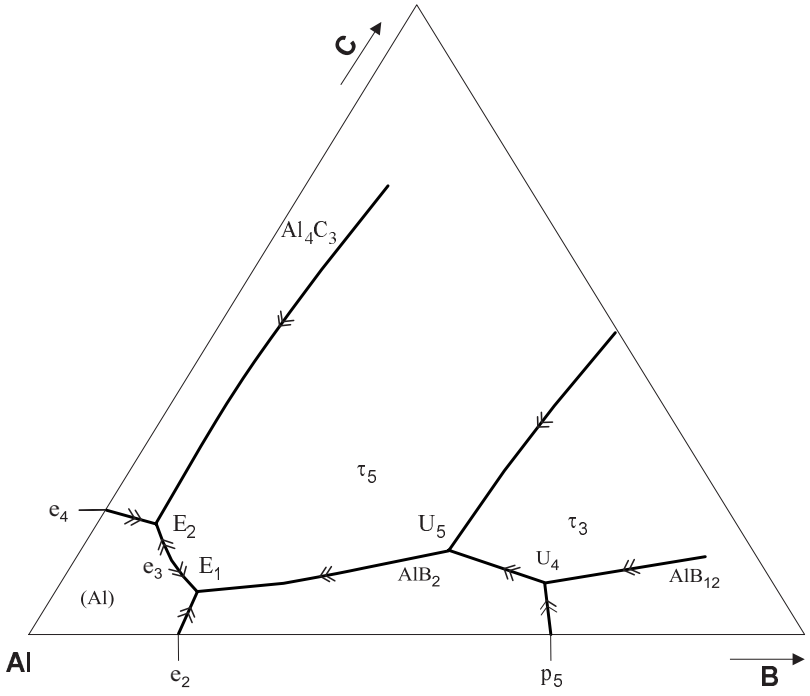

Fig. 6: Al-B-C.
Tentative liquidus surface projection

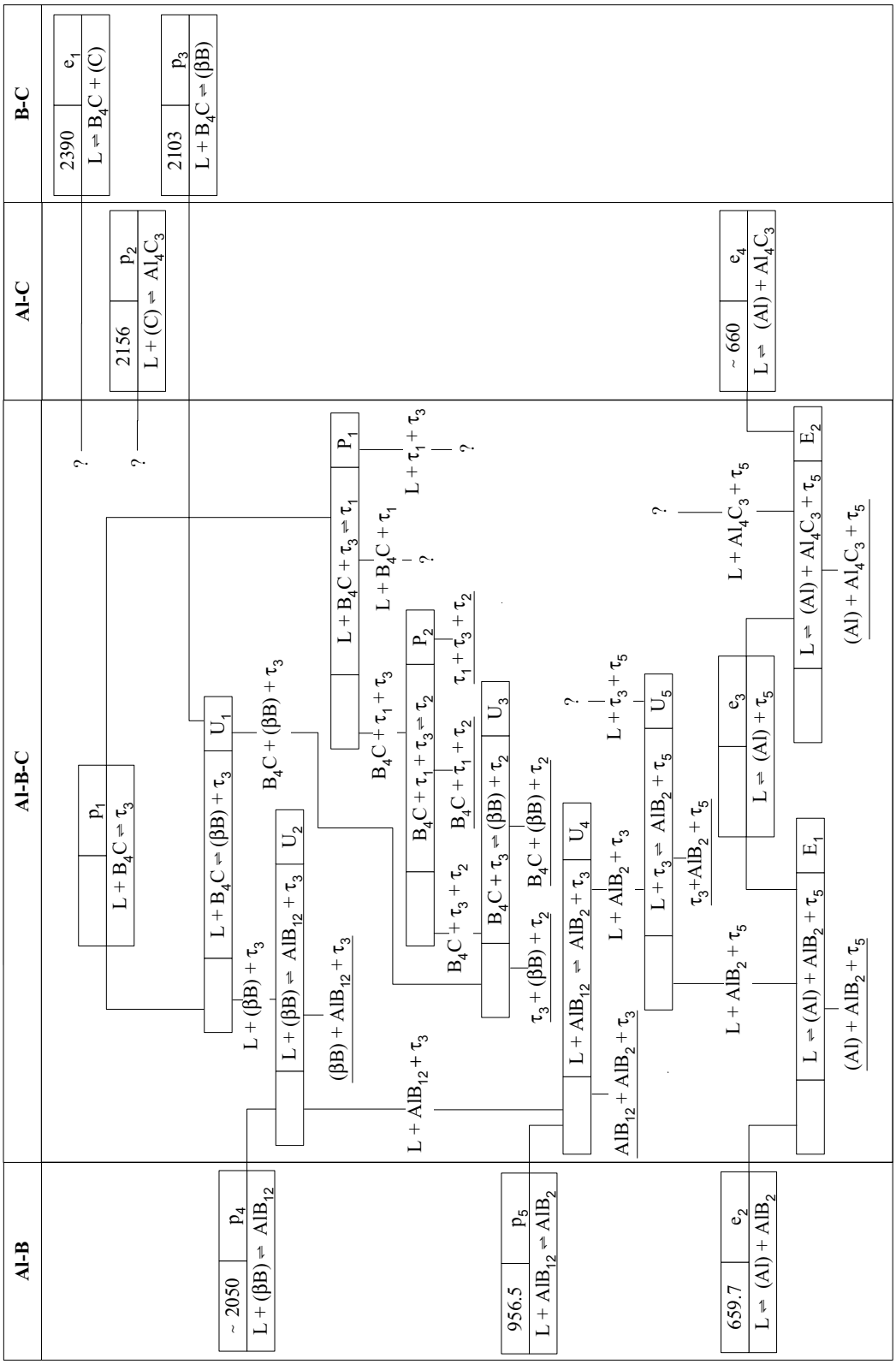

Fig. 7: Al-B-C. Reaction scheme