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Short-range order in undercooled and stable melts forming quasicrystals and approximants and its influence on nucleation

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Abstract

An icosahedral short-range order has been predicted to prevail in undercooled metallic melts. For melts of pure metals this hypothesis was recently experimentally confirmed by diffraction experiments.

This work presents results of neutron scattering experiments on the short-range order of stable and undercooled liquids of alloys forming quasicrystals and polytetrahedral crystals. The studies indicate that an icosahedral topological short-range order prevails in all of the investigated melts. The influence of this icosahedral short-range order on the nucleation behaviour of solid phases from the undercooled liquid is discussed.

Keywords: undercooling, short-range order, quasicrystals, nucleation, solid-liquid interfacial energy

1. Introduction

Following a consideration of Frank [1], an icosahedral short-range order (ISRO) should be energetically favourable in undercooled melts of systems consisting of atoms of roughly spherelike geometrical symmetry such as metals. An icosahedron is a polytetrahedron built up from 13 atoms. It is characterized by six fivefold symmetry axes. If atomic interactions according to a Lennard-Jones potential are assumed, the energy of an icosahedral cluster is about 8.4% smaller than that of 13 atoms clusters with fcc- or hcp-structure. While this argument is concerned with small clusters, later the idea of a polytetrahedral short-range order (SRO) in undercooled melts was confirmed also for larger systems by molecular dynamics computer simulations on Lennard-Jones liquids [2,3], which revealed an increasing degree of ISRO with increasing undercooling, ΔT , of the melt.

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3 While Frank's hypothesis of an icosahedral short-range order dates from 1952, from
4 the experimental side the question of the short-range order in undercooled metallic melts
5 remained unsolved for about fifty years. In 2000 the structure of liquid Pb on a Si (001)
6 substrate was studied at the solid (Si) -liquid (Pb) interface by X-ray diffraction at
7 temperatures, T , above the liquidus temperature, T_L [4]. The experiments provide evidence of
8 a fivefold local symmetry of the liquid lead at the Si(001) interface and thus ISRO. In these
9 experiments, however, the SRO was not studied in the bulk liquid.

10
11 Recently, we have investigated the SRO of bulk liquids of the pure metals Fe, Zr, Ni
12 and Co in the stable and deeply undercooled regime by diffraction of neutron- and
13 synchrotron radiation [5,6]. The melts were undercooled using the containerless processing
14 technique of electromagnetic levitation. These studies provide a direct experimental proof of
15 an ISRO prevailing all examined bulk metallic melts, independent on the structure of the
16 corresponding solid phase. The ISRO becomes more pronounced at lower temperatures.

17
18 By Lee et al. [7] the SRO of stable and undercooled liquid Ni and Ti was investigated
19 by combination of the undercooling technique of electrostatic levitation with diffraction of
20 synchrotron radiation. This work confirms our results on liquid Ni. For melts of Ti
21 indications for distortions of the ISRO are reported.

22
23 The above mentioned studies are concerned with monoatomic systems. For alloys,
24 despite aspects of the topological SRO also those of a possible chemical order must be
25 considered. Moreover, if an alloy consists of atoms with a large difference in the atomic
26 radii, even a topological SRO different from ISRO may be favoured for geometric reasons.
27 Nevertheless, as long as the atomic radii of the alloy components are close, an ISRO will still
28 be advantageous. A small concentration of slightly smaller or larger atoms may even stabilize
29 icosahedral clusters because the interatomic distance between the atoms on the surface of an
30 icosahedron is approximately 5% larger than the distance between the surface atoms and the
31 central atom [8]. Hence, placing a smaller atom into the center of an icosahedron or larger
32 atoms on its surface is energetically favourable.

33
34 Jónsson and Andersen [9] investigated a binary Lennard-Jones liquid model consisting
35 of 1500 atoms. 20% of these atoms were 25% larger than the other species. In the deeply
36 undercooled liquid, 138 interpenetrating and interconnected icosahedra were found, such that
37 61% of all atoms were within an icosahedral environment.

38
39 Of special scientific interest is the investigation of the SRO of alloy melts that form
40 quasicrystalline or the structurally closely related crystalline approximant phases. These
41 phases are characterized by ISRO in the solid state.

42
43 At temperatures above T_L the SRO of the quasicrystal-forming alloy $Al_{80}Mn_{20}$ [10]
44 was studied by neutron scattering. Atomic pair potentials have been inferred from these
45 experiments, which were subsequently used for molecular dynamics calculations [11]. These
46 calculations revealed an icosahedral ordering even in stable melts ($T > T_L$), while in the
47 undercooled regime ($T=0.8 \cdot T_L$) this SRO is even more pronounced.

48
49 Neutron diffraction studies on stable quasicrystal-forming Al-Pd-Mn melts delivered
50 structure factors, $S(Q)$, which could be simulated under the assumption that icosahedral
51 clusters with Mn- or Pd-atoms located in the centre of the icosahedron [12] prevail in the
52 liquid. For other SROs, for instance those of hcp or fcc symmetry, the calculated $S(Q)$ do
53 not deliver such a good fit of the experimental results.

54
55 This paper presents the results of our recent neutron diffraction investigations on the
56 SRO of undercooled melts of Al-based alloys forming quasicrystals and approximants
57 ($Al_{60}Cu_{34}Fe_6$, $Al_{65}Cu_{25}Co_{10}$ and $Al_{13}(Co,Fe)_4$). The impact of the SRO in the melt on the
58 nucleation behaviour of solid phases of different structure is discussed. All of the
59 quasicrystal-forming alloy systems studied here possess only a moderate difference of the
60

atomic radii. Hence, an ISRO should be favourable in the liquid state from the theoretical point of view.

2. Short-range order in melts forming quasicrystals and approximants

In order to deeply undercool melts, heterogeneous nucleation at foreign phases such as container walls or impurity sites must be suppressed [13]. Therefore, the melts are containerlessly processed under high purity conditions by use of the electromagnetic levitation technique [13]. Elastic neutron scattering experiments were performed on the electromagnetically levitated samples approximately 1 g in mass at the high-intensity two-axis diffractometer D20 of the Institut-Laue-Langevin (ILL) in Grenoble, France. Experimental setup and data treatment are described in ref. [14].

Figure 1 shows the structure factor, $S(Q)$, of $\text{Al}_{60}\text{Cu}_{34}\text{Fe}_6$ melts forming an icosahedral quasicrystalline I-phase that was determined by neutron diffraction at different temperatures ($T = 1090$ K, $T = 1225$ K and $T = 1325$ K) above and below the liquidus temperature of $T_L = 1164$ K. The $S(Q)$ of the $\text{Al}_{60}\text{Cu}_{34}\text{Fe}_6$ melts exhibit a shoulder on the right side of the second oscillation. Moreover, at small Q vectors a prepeak occurs that suggests the existence of a medium-range order. Both features of $S(Q)$ become better defined, if T is decreased.

A similar shape of $S(Q)$ was observed in our neutron scattering investigations on $\text{Al}_{65}\text{Cu}_{25}\text{Co}_{10}$ melts forming a decagonal quasicrystalline D-phase [15]. Also diffraction investigations with synchrotron radiation performed by Kelton et al. [16] on $\text{Ti}_{39.5}\text{Zr}_{39.5}\text{Ni}_{21}$ melts forming an I-phase, which have been undercooled by electrostatic levitation, revealed the characteristic shoulder on the second oscillation of $S(Q)$.

A similar shoulder was observed in the $S(Q)$ of monoatomic metallic melts [5,6]. It is considered as a first indication of an icosahedral topological SRO [8]. For alloys, however, this interpretation is not as unambiguous as for monatomic systems due to the fact that the total $S(Q)$ measured for alloys is affected by both the topological and the chemical structure.

In order to separate effects resulting from the chemical SRO from those due to the topological SRO, partial structure factors must be determined. For such investigations we have chosen $\text{Al}_{13}(\text{Co,Fe})_4$ alloys forming approximant phases. **The melting temperatures of the alloys are nearly independent of the Co/Fe ratio (ranging between approximately $T_L = 1420$ K for $\text{Al}_{13}\text{Fe}_4$ and $T_L = 1440$ K for $\text{Al}_{13}\text{Co}_4$).** When assuming an isomorphous substitution of Co by Fe the transition metal atoms (TM) can be considered as one component of a quasi-binary Al-TM alloy and changing the Fe/Co ratio allows to vary the coherent neutron scattering cross section of the TM-component. From at least three neutron scattering examinations with different scattering contrast of the components (Al, TM) the complete set of partial structure factors within the Faber-Ziman formalism [17] and the Bhatia-Thornton formalism [18] can be determined. The Faber-Ziman structure factors $S_{AlAl}(Q)$, $S_{AlTM}(Q)$ and $S_{TMTM}(Q)$ are the contributions to $S(Q)$, which result from the three different kinds of **atomic pairs** (Al-Al, Al-TM and TM-TM). Within the Bhatia-Thornton formalism the partial structure factor $S_{NN}(Q)$ describes exclusively the topological SRO of the system, $S_{CC}(Q)$ the chemical SRO and $S_{NC}(Q)$ the correlation of density and concentration. We measured the total $S(Q)$ of stable and undercooled melts of five different $\text{Al}_{13}(\text{Co,Fe})_4$ alloys, such that the equation system to determine the partial structure factors is overdetermined. **The partial structure factors inferred from one subset of three measurements agree with those inferred from other subsets of data within the statistical error limits that correspond to the spread of the data points.** This allowed us to verify the hypothesis of an

isomorphous substitution of the TM-atoms with hindsight [19].

Figure 2 shows the Faber-Ziman (a) and the Bhatia-Thornton (b) partial structure factors determined from the five neutron scattering measurements for moderately undercooled melts at $T = T_L - 60$ K. The results obtained at $T = T_L - 150$ K are shown in Fig. 2 of ref. [19] and those determined at $T = T_L + 150$ K are depicted in Fig. 2 of ref. [20].

It is noteworthy that at all temperatures $S_{NN}(Q)$ exhibits the characteristic shoulder on the right hand side of the second oscillation indicating that this feature also visible in the $S(Q)$ of the $Al_{60}Cu_{34}Fe_6$ (see figure 1) and $Al_{65}Cu_{25}Co_{10}$ [15] melts results from the topological SRO. Consequently, similar as for melts of pure metals [5,6], this shoulder is an indication of an ISRO prevailing in the liquid [8]. This interpretation is confirmed by simulations of $S_{NN}(Q)$ at large Q vectors by assuming that the melt contains separate tightly bound clusters. The best fit of the experimental results is obtained if an ISRO is assumed to prevail in the liquid [19]. There are indications that this ISRO consists of larger polytetrahedral aggregates such as dodecahedral clusters with 33 atoms.

Apart from this icosahedral topological SRO the analysis of the partial pair correlation functions, which are shown in ref. [19] for all investigated temperatures, indicates the existence of a chemical SRO in $Al_{13}(Co,Fe)_4$ melts, such that the formation of Al-TM nearest neighbours is favoured, while TM-TM nearest neighbours are avoided [19]. This chemical short-range order explains the occurrence of the prepeaks in the total structure factors of $Al_{13}(Co,Fe)_4$, $Al_{60}Cu_{34}Fe_6$ and $Al_{65}Cu_{25}Co_{10}$.

3. Impact of the short-range order on the nucleation behaviour of undercooled liquids

According to classical nucleation theory [21] for the intrinsic case of homogeneous nucleation the steady-state nucleation rate of a solid phase in an undercooled melt is given by:

$$I_{SS} \approx 10^{39} \cdot \exp\left(-\frac{\Delta G^*}{k_B T}\right) [m^{-3}s^{-1}] \quad \text{with} \quad \Delta G^* = \frac{16\pi\gamma^3}{3\Delta G_V} \quad (1)$$

Here k_B denotes Boltzmann's constant, ΔG^* the activation threshold for nucleation, ΔG_V the difference of the Gibbs free energies of liquid and solid and γ the solid-liquid interfacial energy, which will depend on the structure of both the liquid and of the solid.

One model for γ that considers structural aspects and depends only on experimentally measurable parameters is the negentropic model [22,23,24,8]. Here, γ is expressed by:

$$\gamma(T) = \alpha \Delta S_f T (N_L V_m^2)^{-1/3} \quad (2)$$

V_m denotes the molar volume, N_L Avogadro's number and ΔS_f the entropy of fusion per mol. The dimensionless solid-liquid interfacial energy α depends on the structure of the solid. For non complex crystalline phases with fcc, hcp or bcc structure α has been analytically determined [22,23,24,8]. These estimations base upon the assumption of tetrahedral SRO in the solid-liquid interface. This is motivated by Frank's prediction of a polytetrahedral SRO in the melt, which - as shown above - has meanwhile been experimentally verified for metallic systems. Because for structurally complex phases an analytical calculation of γ is

impossible, the negentropic model was recently extended in order to numerically estimate α also for such phases [26,27]. The calculations were performed for different solid structures: the icosahedral quasicrystalline I-phase in Al-Pd-Mn, the polytetrahedral phases λ -Al₁₃Fe₄ and μ -Al₃Fe₂, the σ' -phase in Ni-V, which is a Frank-Kasper phase with a Z14 coordination polyhedron and the hard magnetic phase Φ -Nd₂Fe₁₄B₁. The Φ -phase is a structurally complex solid, but different from the other investigated structures, it is not a polytetrahedral phase. The results of the numerical calculations [26,27] and of the analytical estimations by Spaepen and Thompson [22,23,24,8] are summarized in the following relation:

$$\alpha_{calc}^I \approx 0.34 < \alpha_{calc}^{\sigma'} \approx 0.36 < \alpha_{calc}^{\Phi} \approx 0.37 < \alpha_{calc}^{\lambda} \approx 0.39 < \alpha_{calc}^{\mu} \approx 0.43 < \alpha_{calc}^{bcc} \approx 0.70 < \alpha_{calc}^{fcc/hcp} \approx 0.85. \quad (3)$$

The quasicrystalline I-phase exhibits the lowest value of α , while for the structurally non complex phases with bcc, fcc and hcp structures the largest α -factors are estimated. For the structurally complex phases λ , μ , σ' and Φ remarkably low values of α are predicted. The fact that the Φ -phase is a not a polytetrahedral structure highlights that a small α is not only anticipated for solid phases with a polytetrahedral SRO which resembles that of the liquid phase. The modelling suggests that the geometrical constraints during construction of the solid-liquid interface, which result from structural complexity, give rise to a low α .

Because γ determines ΔG^* in the third power (eq. (1)), the relation (3) of α implies a strong dependence of the maximum undercoolability of metallic melts on the topological structure of the solid phases formed from the undercooled liquids. This structure dependence was systematically studied for Al-based quasicrystal forming liquids undercooled by electromagnetic levitation [25,26,27]. Maximum relative undercoolings of only $\Delta T^I/T_L^I \approx 0.1$ were measured for the I-phases in Al-Cu-Fe and Al-Pd-Mn, while for the decagonal quasicrystalline D-phases in Al-Cu-Co, Al-Ni-Co and Al-Co $\Delta T^D/T_L^D \approx 0.15$ is obtained. $\Delta T^{\lambda}/T_L^{\lambda} \approx 0.13$ was observed for the λ -phase in Al(-Cu)-Fe and $\Delta T^{\mu}/T_L^{\mu} \approx 0.14$ for the μ -phase in Al-Fe. The crystalline β -phase in Al-Cu-Co of CsCl structure (topological structure equivalent to a bcc phase) exhibits the largest undercoolability ($\Delta T^{\beta}/T_L^{\beta} \approx 0.25$).

In order to estimate α from the measured undercoolings, ΔT , we assume homogeneous nucleation and that at least one nucleation event occurs during the experiment time t_N within the sample volume, V , for the onset of solidification at the nucleation temperature T_N : $I_{SS}(T_N) \cdot V \cdot t_N \geq 1$. With eq. (1) and (2) the dimensionless solid-liquid interfacial energy, α , can be estimated from experimental results on the maximum undercoolability under assumption of homogeneous nucleation, because all other parameters are measurable or can be estimated by models. As described in detail in ref. [26], this gives a similar sequence of α as relation (3):

$$\alpha^I \approx 0.3 < \alpha^{\lambda} \approx 0.33 < \alpha^{\mu} \approx 0.37 < \alpha^D \approx 0.45 < \alpha^{\beta} \approx 0.63 \quad (4)$$

The structure dependence of γ has a decisive impact on the phase selection during solidification of undercooled melts. Although $\Delta G_{\gamma}(T)$ is always lower for stable solid phases than for metastable ones, at a given $T < T_L$, $\Delta G^*(T)$ may be smaller for a metastable phase, if this phase has a sufficiently lower $\gamma(T)$, resulting in the nucleation of metastable phases.

For example, under equilibrium conditions Al₆₀Cu₃₄Fe₆ melts form the λ -phase at the liquidus temperature of $T_L^{\lambda}=1164$ K and, in a second solidification step, the I-phase is peritectically formed from λ and the liquid at $T_p^I=1115$ K [28]. If, however, the melt is undercooled deeply enough, the I-phase crystallizes primarily [26,28]. This phase selection behaviour is consistent with relation (3) which suggests that γ and therefore also ΔG^* is lower for the I-phase than for the λ -phase. It must be stressed that our neutron scattering

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3 experiments on $\text{Al}_{60}\text{Cu}_{34}\text{Fe}_6$ melts (figure 1) are indicative of an ISRO, such that a direct link
4 between the SRO in the liquid phase and the undercooling and phase selection behaviour has
5 been established for this alloy. Also the phase selection properties of other undercooled melts
6 of Al-Cu-Fe alloys such as $\text{Al}_{62}\text{Cu}_{25.5}\text{Fe}_{12.5}$ can be explained by the structure dependence of
7 γ as predicted by relation (3) [28].
8
9

10 4. Conclusions

11
12 The SRO of undercooled and stable melts forming quasicrystals and their approximants
13 ($\text{Al}_{60}\text{Cu}_{34}\text{Fe}_6$, $\text{Al}_{65}\text{Cu}_{25}\text{Co}_{10}$ and $\text{Al}_{13}(\text{Co,Fe})_4$) was studied by neutron scattering. For the
14 $\text{Al}_{13}(\text{Co,Fe})_4$ melts partial Faber-Ziman- and Bhatia-Thornton structure factors were
15 determined. The experiments provide evidence of an icosahedral topological short-range order
16 prevailing in the melts even at temperatures above the melting temperature. There are
17 indications that this SRO consists not only of simple icosahedral clusters with 13 atoms but
18 of larger polytetrahedral aggregates such as dodecahedra. Similar results were obtained from
19 diffraction studies on melts of several pure metals [5,6]. This suggests that ISRO is a
20 universal property of most metallic melts independent of the structure of the corresponding
21 solid phases, provided that the atomic radii of the components are not too different.
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23

24
25 The energy of the interface between a solid nucleus and an undercooled melt was
26 modeled in the framework of the negentropic model. This model bases on the assumption of
27 a polytetrahedral SRO in the solid-liquid interface as proven to exist in metallic melts by our
28 diffraction studies. A pronounced dependence of the solid-liquid interfacial energy on the
29 structure of the solid phase is predicted by the model. This structure dependence is able to
30 explain the undercooling behaviour of melts of Al-based alloys forming solid phases of
31 different structure as well as the phase selection behaviour during solidification.
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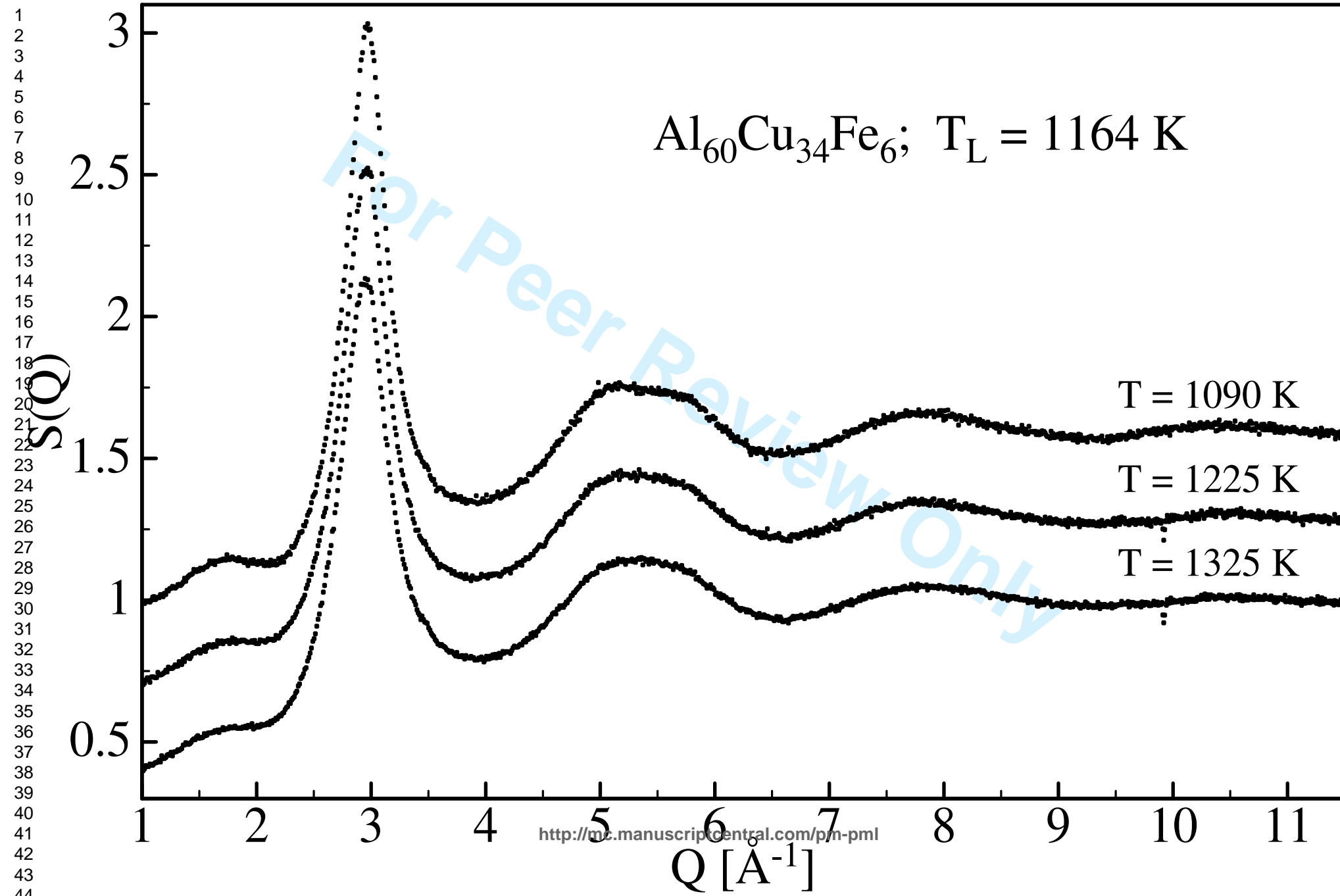
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