

# Time-of-flight neutron-scattering study of phason hopping in decagonal Al-Co-Ni quasicrystals

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We have measured phason hopping in isotopic powder samples of decagonal Al-Co-Ni quasicrystals, by time-of-flight quasielastic neutron scattering. The three isotopic samples were based on  $^{nat}\text{Ni}$ , and 99.5% enriched  $^{58}\text{Ni}$  and  $^{60}\text{Ni}$ . Two characteristic jump times have been observed. The anomalous temperature dependence observed in the icosahedral phases *i*-Al-Cu-Fe and Al-Mn-Pd is confirmed.

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## I. INTRODUCTION

In quasicrystals (QC's) atomic hopping (so-called phason dynamics) <sup>1-5</sup> has a bearing on such varied issues as stability (the random-tiling model),<sup>6</sup> diffusion,<sup>7</sup> growth,<sup>8</sup> phase transitions between QC and related phases,<sup>9</sup> high-temperature superplasticity,<sup>10</sup> and cross-checking structural models.<sup>11</sup> It has been studied extensively,<sup>1-4</sup> by quasielastic neutron scattering and by  $^{57}\text{Fe}$  Mössbauer spectroscopy. Mainly two samples have been studied, viz., the icosahedral phases *i*-Al<sub>62</sub>Cu<sub>25.5</sub>Fe<sub>12.5</sub> and *i*-Al<sub>70.3</sub>Pd<sub>21.4</sub>Mn<sub>8.3</sub>. Both alloys serve as important standard reference probes as they are stable at room temperature and can be obtained in large volumes as required for neutron-scattering studies. Whereas large single-grain samples of *i*-Al-Mn-Pd can be grown by the Czochralsky method, this is not the case for *i*-Al-Cu-Fe as its stability range in the *i*-Al-Cu-Fe phase diagram is not in contact with the liquidus, such that only polycrystalline samples can be obtained by the melt-spinning technique. Nevertheless within this system isotopic substitutions (of the Fe and Cu atoms) are feasible that result in good neutron-scattering contrasts and are reasonably cheap. In *i*-Al<sub>70.3</sub>Pd<sub>21.4</sub>Mn<sub>8.3</sub>, such isotopic contrasts are poor and the isotopes are expensive. Hence the two systems are complementary. In the study of *i*-Al-Cu-Fe one can assess the species of the jumping atoms, while in *i*-Al-Mn-Pd one can get information on the jump vectors.

Whereas all previous studies were limited to *icosahedral* phases, here we are reporting neutron scattering results on phason hopping within a *decagonal* QC, viz., *d*-Al-Co-Ni. In addition to its purely scientific interest, this project also presents a number of technical advantages. In fact, the accurate determination of the jump vectors and their abundance in a single-grain of *i*-Al-Mn-Pd is somewhat hampered by the high symmetry of the icosahedral point group.<sup>12</sup> At low *Q* values the quasielastic signals are highly isotropic. In the *decagonal* QC *d*-Al-Co-Ni the effects could be more marked. The two-dimensional character of the quasiperiodicity may give rise to pronounced anisotropies here. Moreover, isotopic substitutions ( $^{58}\text{Ni}$  and  $^{60}\text{Ni}$ ) are feasible<sup>13</sup> and large single-grain samples can be produced by floating-zone<sup>14</sup> or flux-growth methods.<sup>15</sup> The first phonon studies on such single-grain samples have been reported.<sup>16</sup>

## II. EXPERIMENT

The scope of the present time-of-flight (TOF) study on powder samples has been dictated by a concern to achieve the best complementarity with a triple-axis study on a large single-grain sample that is in progress. Consequently, the emphasis is put on the four following aspects. (1) The temperature dependence. (2) Isotopic substitutions [as the growth of large isotopic single-grain samples remains a prohibitively expensive nirvana, that would require vast quantities (several tens of grams)<sup>17</sup> of isotopes]. (3) The widths of the Lorentzian signals, which can in principle be determined with a better precision on a TOF spectrometer such as MIBEMOL (which has a triangular resolution function) than on a triple-axis spectrometer. (4) A preparation of the triple-axis study by establishing the list of areas in reciprocal space of interest, and by defining the best parameters for the scans.<sup>12</sup>

The samples were prepared by arc-melting (Degussa VOlI 0) compacts of the mixed elemental powders (Haereus: Al 99.95%, natural Ni 99.99%; Chempur: Co 99.99%; Eurisotope:<sup>18</sup>  $^{58}\text{Ni}$  99.94%,  $^{60}\text{Ni}$  99.6%) with starting composition Al<sub>71</sub>Co<sub>12</sub>Ni<sub>17</sub>, for natural Ni and Al<sub>71.5</sub>Co<sub>13</sub>Ni<sub>15.5</sub> for the pure isotopes  $^{58}\text{Ni}$  and  $^{60}\text{Ni}$ . Then the ingots were molten in a high-vacuum furnace (PVA,  $1 \times 10^{-4}$  Pa) and annealed for 29 h at 900 °C under an argon pressure of 50 kPa. Subsequently, the samples were quenched by jetting cold argon in the sample chamber of the switched-off furnace. The total weight loss during sample preparation ran up to approximately 1%.

The TOF neutron-scattering experiments have been done using the spectrometer MIBEMOL at the Orphée reactor of the Laboratoire Léon Brillouin in Saclay, France. The 434 <sup>3</sup>He detectors of the instrument were grouped in ten angular banks centered at  $2\theta$  positions of 25.5, 40.1, 47.1, 55.1, 63.1, 75., 89.9, 105.3, 151.3, 137.4 degrees. In a first series of experiments 10.622 grams of powder of the so-called Edagawa phase<sup>19</sup> Al<sub>71</sub>Co<sub>12</sub>Ni<sub>17</sub> with a natural isotopic composition were put into a cylindrical alumina holder mounted within a cylindrical thin-walled Nb frame. The whole was hung inside a furnace within the neutron beam. Data were collected with incident neutron wavelengths  $\lambda$  of 4, 5, and 8 Å. The corresponding elastic resolutions  $\Delta E$  are 310, 159, and 39  $\mu\text{eV}$  [full width at half maximum (FWHM)] respec-

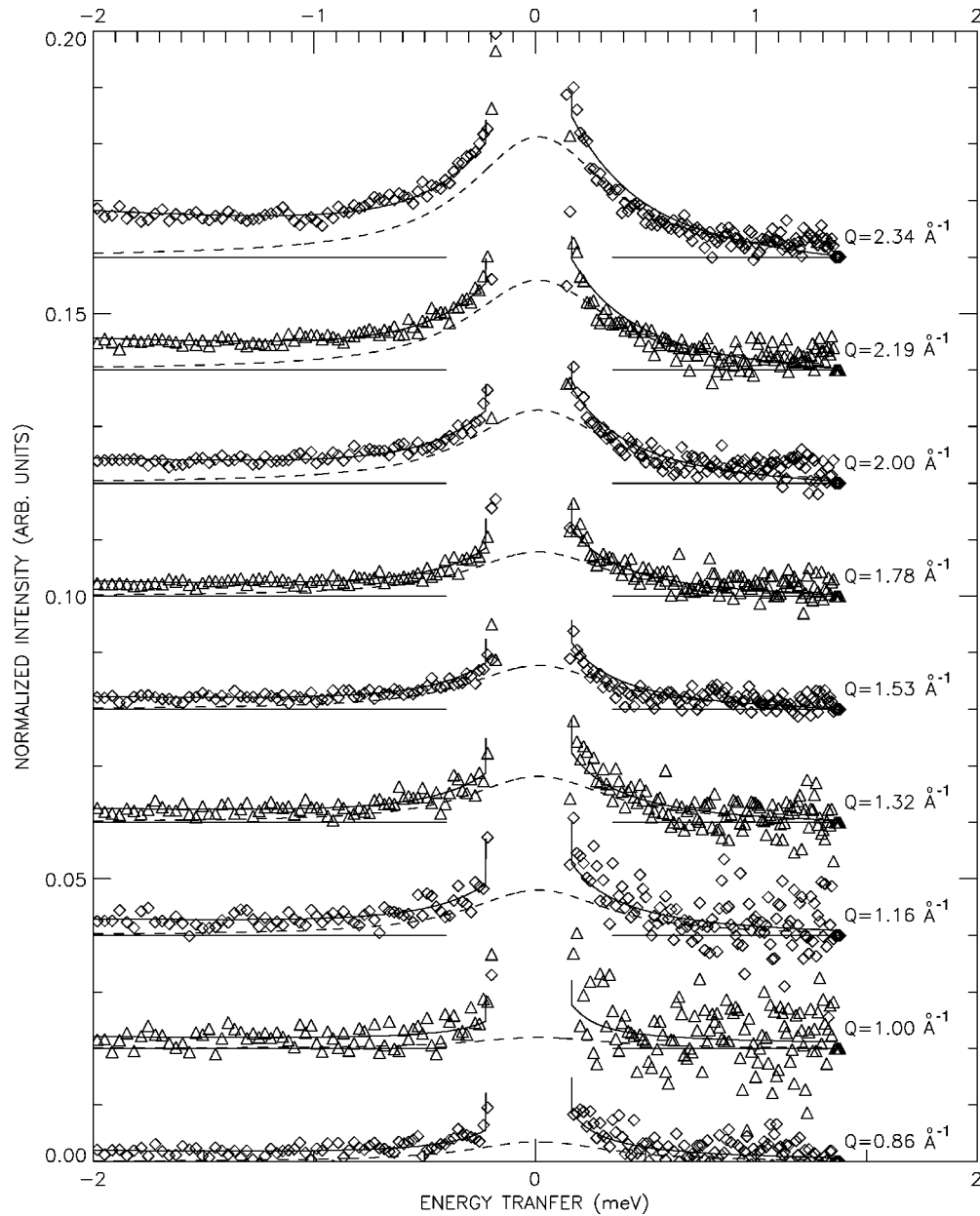


FIG. 1.  $Q$  dependence of the (background-corrected)  $\lambda = 5$  Å spectra for  $\text{Al}_{71}\text{Ni}_{17}\text{Co}_{12}$  at  $940^\circ\text{C}$ . The total fits and their Lorentzian components are also shown.

tively. The sample was measured at room temperature,  $500$ ,  $650$ ,  $700$ ,  $780$ ,  $820$ ,  $862$ ,  $900$ ,  $940^\circ\text{C}$  in the  $5$  Å runs, and at  $650$ ,  $800$ ,  $936^\circ\text{C}$ , in the  $4$  Å runs. The  $8$  Å run at  $936^\circ\text{C}$  did not show quasielastic broadening and will accordingly not be discussed any further. (There is thus no quasielastic intensity in this  $Q$  range on the timescale given by  $\hbar/\Delta E$ . The signal seen in the  $\lambda = 5$  Å data is too wide with respect to the elastic resolution  $\Delta E = 39$   $\mu\text{eV}$  to be observable in the  $\lambda = 8$  Å run.) Empty-container-assembly runs were made at room temperature and  $700^\circ\text{C}$  ( $\lambda = 5$  Å) and at  $700^\circ\text{C}$  ( $\lambda = 4$  Å). A vanadium normalization run at room temperature ( $\lambda = 5$  Å) completed the data set. In a second series the sample of the natural isotopic composition was measured again, together with samples that were entirely enriched in  $\text{Ni}^{58}$  ( $7.43$  g) and in  $\text{Ni}^{60}$  ( $7.236$  g). The measurements were done with incident wavelengths  $\lambda$  of  $4$  and  $5$  Å at temperatures of  $940$  and  $650^\circ\text{C}$ . Some additional runs at  $850^\circ\text{C}$  were also made

( $\text{Ni}^{58}$  and natural Ni with  $\lambda = 5$  Å; natural Ni with  $\lambda = 4$  Å.) The empty cell was measured at  $940$  and  $650^\circ\text{C}$  and the vanadium sample at room temperature. These background and normalization runs were only made with  $\lambda = 5$  Å incoming neutrons. Typical acquisition times were of the order of one day per run in the first series and two days in the second series of experiments. The choice of the temperatures for the background runs is based on our knowledge of the experimental device, which is the same as has been used in the experiments described in Ref. 2, where more technical details on the specific point of the background characteristics can be found. The main point for us is that the background is *flat* in the energy region of interest, and not varying much with temperature, such that a more elaborate determination of it would be superfluous. Self-shielding and multiple scattering effects are negligible in our experiments. After the usual operations for background correction and vanadium

normalization<sup>1,2</sup> the  $\lambda=5$  Å data were fitted by a three-component expression

$$\left[ A_0(Q) \delta(\omega) + A_1(Q) \frac{1}{\pi} \frac{\Gamma}{\omega^2 + \Gamma^2} + A_2(Q, \omega) \right] \otimes \mathcal{R}(Q, \omega), \quad (1)$$

where  $\mathcal{R}(Q, \omega)$  is the instrument resolution function obtained from fits of the vanadium run. The Dirac measure describes the elastic scattering.  $A_2(Q, \omega) \propto e^{-\langle u^2 \rangle Q^2} Q^2 g(\omega) n(\omega, T) / \omega^2$  is a linear term and it is essential in order to take into account the background of the low-energy phonon density of states.

### III. RESULTS AND DISCUSSION

The detailed fit procedures and their inherent difficulties have been discussed many times elsewhere. The results for the natural-nickel-based sample are summarized in Figs. 1–4. and can be described as follows. The  $\lambda=5$  Å data exhibit a quasielastic line with a width  $\Gamma(Q)$  that within the limits of the experimental precision is constant with  $Q$ . Summing up the signals from all the detectors within the range  $Q_{el} \in [1.11, 2.37] \text{ \AA}^{-1}$ , the fit gives a value  $\Gamma \approx 365 \mu\text{eV}$ , which we further fixed in the analysis of the  $Q$  dependence of the quasielastic intensity  $\mathcal{J}$ . This  $Q$  dependence is reported in Figs. 1,2 for the 940 °C data. We see that the quasielastic structure factor does not reach its first maximum below  $Q = 2.37 \text{ \AA}^{-1}$ , such that the jump distance must be smaller than  $2.0 \text{ \AA}$ . The  $T$  dependence exhibits the same characteristics as has been observed previously for *i*-Al-Cu-Fe and *i*-Al-Mn-Pd.

The intensity  $\mathcal{J}$  of the Lorentzian, quasielastic hopping signal exhibits an Arrhenius-type law (with an activation energy  $E_a \approx 553 \text{ meV}$ ), while its width  $\Gamma$  is practically constant with temperature (see Figs. 3,4). This is the opposite of the conventionally observed temperature dependence, where it is  $\Gamma$  that shows an Arrhenius-like behavior and  $\mathcal{J}$  that remains constant with  $T$ . This unusual behavior has been modeled and interpreted in terms of assisted hopping in Ref. 20. The main idea behind this model can be sketched as follows. The energy barrier of the double-well potential is so high that the atoms are, practically speaking, not apt to jump within the time scales we are dealing with here. Of course they are apt to jump on much longer time scales but these cannot be assessed with the energy resolution available here. Hence the abundance of atoms that are apt to jump (on the time scales dealt with in this paper) is in principle zero. If we assume that there is another process that changes the whole environment to one where jumps become observable, then the energy cost to produce this environment will enter into the Boltzmann factor for the number of good environments, i.e., the number of atoms that are apt to jump. This could, e.g., be as follows. A thermal vacancy could come along. The jumping atom would not jump into this vacancy, but the vacancy would sufficiently loosen the environment to make the jump possible. The important point is that the atom does not jump into the vacancy, since else we would get into a normal temperature dependence. There is an alternative to come to an

intensity that changes with  $T$ . That is, the atom would jump inside an asymmetric double well. Both alternatives occur in QC. This will be further discussed in a future publication.

The melting temperature of the sample is around 1125 °C. The decagonal phase, however, is stable only up to approximately 975 °C. At higher temperatures a cubic phase is formed. Our sample should be similar to the sample shown in Fig. 4 of the paper by Baumgarte *et al.*<sup>21</sup> It should be noted that between 717 and 787 °C the satellites labeled S2 by Edagawa become gradually weaker and eventually disappear. Our data point at 780 °C is situated within this region of the Edagawa superstructure. Our data points at 820, 862, and 900 °C are situated within an intermediate region up to 917 °C. At the latter temperature, the S1-type satellites also disappear. Finally, our data point at 940 °C falls into the stability range of the basic decagonal phase. Hence we can observe that with rising temperature our sample goes through all the various phases described in Refs. 19,21, but there is no sign of an accident in the temperature dependence of the quasielastic signal. This finding corroborates our results obtained on the transition between the icosahedral quasicrystalline and the rhombohedral microcrystalline phases within the alloy  $\text{Al}_{62.5}\text{Cu}_{26.5}\text{Fe}_{11}$ . There, also, no discontinuities in the  $T$  dependence of the quasielastic parameters were seen to accompany the structural changes. These facts have a simple explanation<sup>5</sup>: the fast hopping process is determined by the *local* order within the QC.

As for *i*-Al-Mn-Pd (Ref. 2) the  $\lambda=4$  Å TOF data are almost intractable due to the presence of a large phonon background, exemplified in Fig. 5. However by subtracting the 800 °C data from the 936 °C data we could eliminate most of this phonon background and the fits became possible, as demonstrated in Fig. 6 at  $Q=2.74 \text{ \AA}^{-1}$  (which is the data set which is the least affected by the phonon problem). That this procedure works is due to the large value of the assistance energy  $E_a$ : Consequently, the quasielastic signal increases substantially over a temperature interval that entails only minor changes in the phonon density of states. We excluded the elastic peak from the fits as usual.<sup>2</sup> We could determine this way a width  $\Gamma=647 \pm 160 \mu\text{eV}$  at  $Q=2.92 \text{ \AA}^{-1}$ ,  $\Gamma=617 \pm 138 \mu\text{eV}$  at  $Q=2.74 \text{ \AA}^{-1}$ , and

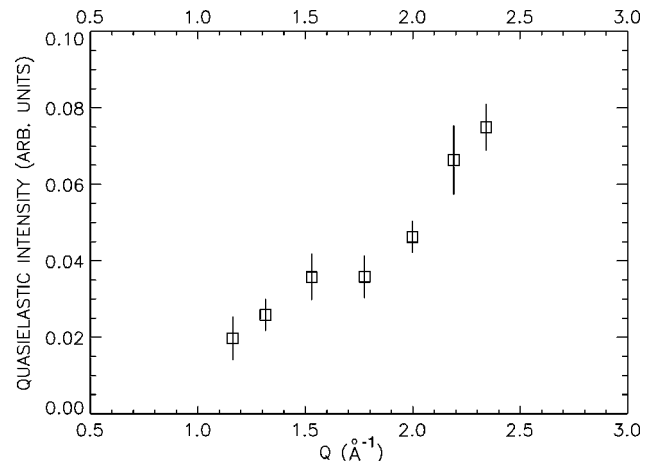


FIG. 2.  $Q$  dependence of the quasielastic intensity  $\mathcal{J}$  in  $\text{Al}_{71}\text{Co}_{12}\text{Ni}_{17}$  obtained from the analysis of the  $\lambda=5$  Å run at 940 °C. The width  $\Gamma(Q)$  is within the limits of precision constant with  $Q$  and is not shown.

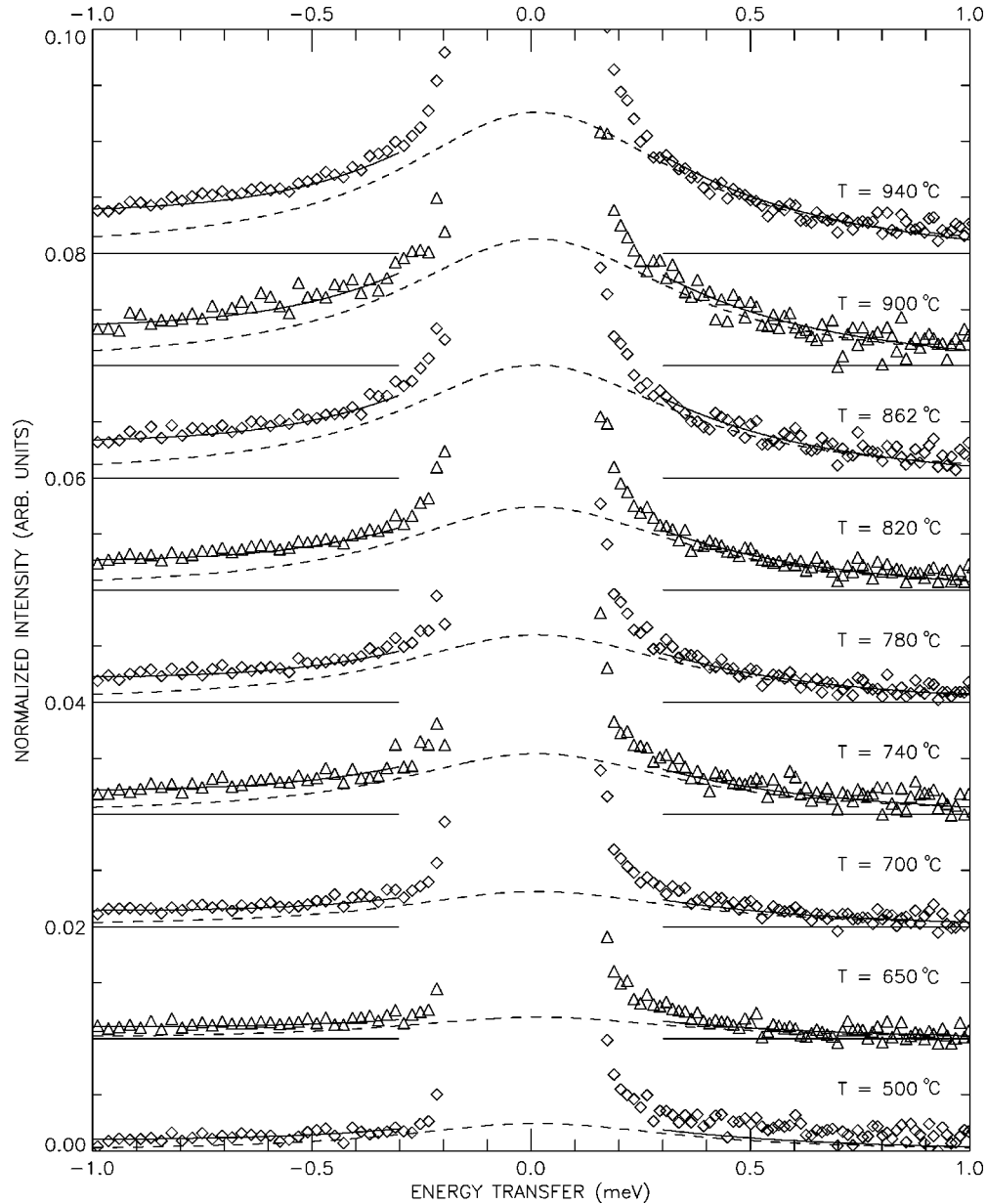


FIG. 3. Temperature dependence of the quasielastic spectra in  $\text{Al}_{71}\text{Co}_{12}\text{Ni}_{17}$  obtained from the analysis of the  $\lambda = 5 \text{ \AA}$  runs. The signals from all detectors with  $Q_{ei} > 1.2 \text{ \AA}^{-1}$  have been summed. The total fits and their Lorentzian components are also shown.

$\Gamma = 413 \pm 122 \text{ \mu eV}$  at  $Q = 2.50 \text{ \AA}^{-1}$ . The latter value is in agreement with the  $\lambda = 5 \text{ \AA}$  result. No temperature or  $Q$  dependence could be determined of the quasielastic signals for  $Q > 2.5 \text{ \AA}^{-1}$ . The scales of Figs. 5 and 6 are directly comparable, which must enable the reader to get an inkling of the inherent difficulties, which are not due to the experimental setup but to the dynamics within the sample itself. Our subtraction procedure must thus rather be considered as a last resort to squeeze some ultimate pieces of (qualitative) information out of our data. The increase of the width  $\Gamma(Q)$  is reminiscent of our findings in  $i\text{-Al-Cu-Fe}$  and  $i\text{-Al-Mn-Pd}$ . Most probably, the quasielastic signal consists of several Lorentzians with widths  $\Gamma_i(Q)$  that are all constant with  $Q$  but with structure factors  $\mathcal{J}_i(Q)$  that vary with  $Q$ , such that the average value of the width  $\langle \Gamma \rangle(Q)$  seems to vary continuously with  $Q$ .

For the isotopic studies it is important to remind the reader that the neutron-scattering contrast between the two Ni isotopes used is very strong: Whereas  $\sigma_{\text{tot}}$  is 26.1 b for  $\text{Ni}^{58}$  it is 0.99 b for  $\text{Ni}^{60}$ . This is a very efficient contrast. The two samples were of the same mass and were measured under strictly identical conditions. Figures 7, 8 show the comparison of the quasielastic signals obtained in the two experimental setups ( $\lambda = 4$  and  $5 \text{ \AA}$ ). By multiplying the intensity of the  $\text{Ni}^{60}$  sample by 1.2 the intensities coincide. This indicates that the signal observed does not imply Ni dynamics. That the signals are not strictly identical might be due to the cumulated effect of various small imperfections in sample alignment and composition, temperature regulation, or to a very small participation of Ni in the jump signal.<sup>22</sup>

The ordering processes occurring in the temperature range studied comprise a global rearrangement of the  $20 \text{ \AA}$  clusters

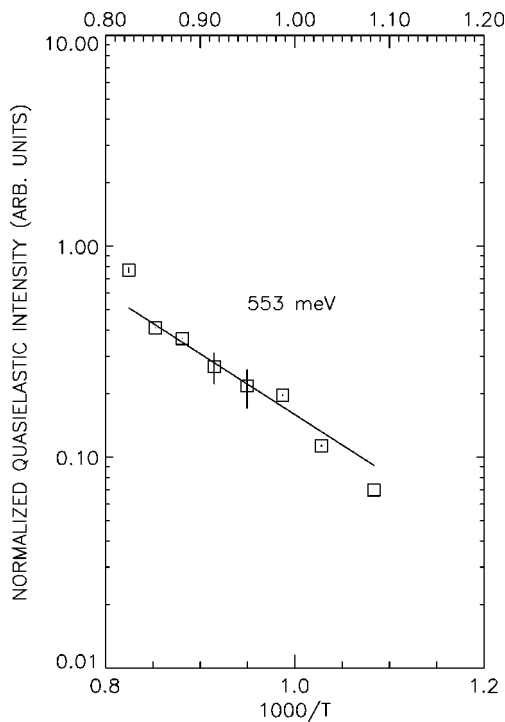


FIG. 4. Arrhenius-like plot of the quasielastic intensity  $\mathcal{J}$  as a function of temperature in the  $\lambda=5$  Å data for  $\text{Al}_{71}\text{Co}_{12}\text{Ni}_{17}$ . The assisting energy is  $\approx 553$  meV.

and connected therewith a chemical ordering between aluminum and transition metal (TM) atoms. Related to the five-dimensional unit cell of the high-temperature basic decagonal phase, the translation period is doubled from approximately four to eight angstroms, and additionally the unit-cell volume is increased by a factor of 5.<sup>23</sup> The basic HT decagonal phase can be interpreted as a cluster-decorated pentagonal tiling which at lower temperature transforms into rhomb-tiling-based superstructure domains of five different orientations.<sup>24</sup> The rearrangement of the 20 Å clusters is performed mainly by phason flips of a few aluminum atoms per

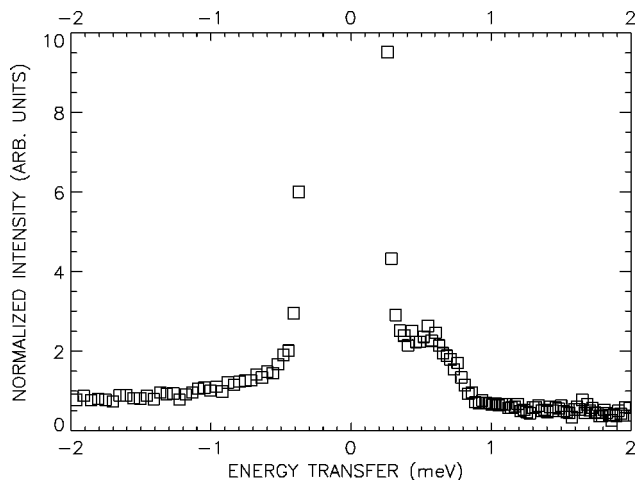


FIG. 5. Typical  $\lambda=4$  Å spectrum of  $\text{Al}_{71}\text{Co}_{12}\text{Ni}_{17}$  at 936 °C in order to show the problem of coherent phonon background. The present data set is obtained by subtracting an empty can run;  $Q_{el}=2.50$  Å<sup>-1</sup>.

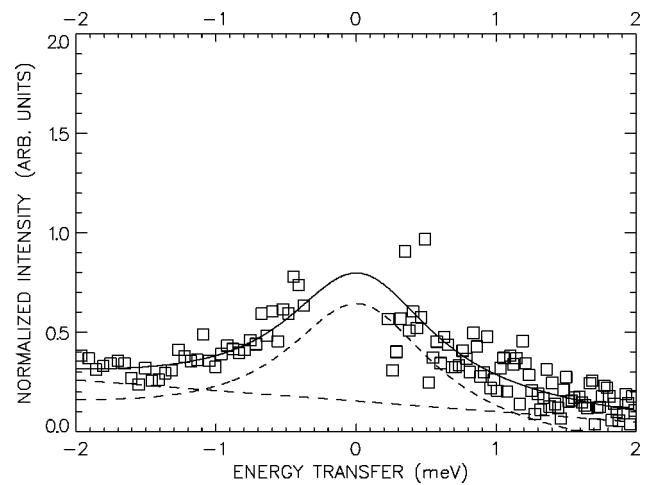


FIG. 6. By subtraction of the 800 °C run from the 936 °C run in the  $\lambda=4$  Å data on  $\text{Al}_{71}\text{Co}_{12}\text{Ni}_{17}$ , the phonon background can be almost entirely eliminated, such that a fit of the quasielastic signal becomes feasible. The spectrum shown corresponds to  $Q_{el}=2.74$  Å<sup>-1</sup>.

cluster (see Fig. 9) with a typical jump distance of 0.938 Å. According to present structure models flips of transition-metal atoms are not very probable. This is also corroborated by simulations based on molecular dynamical

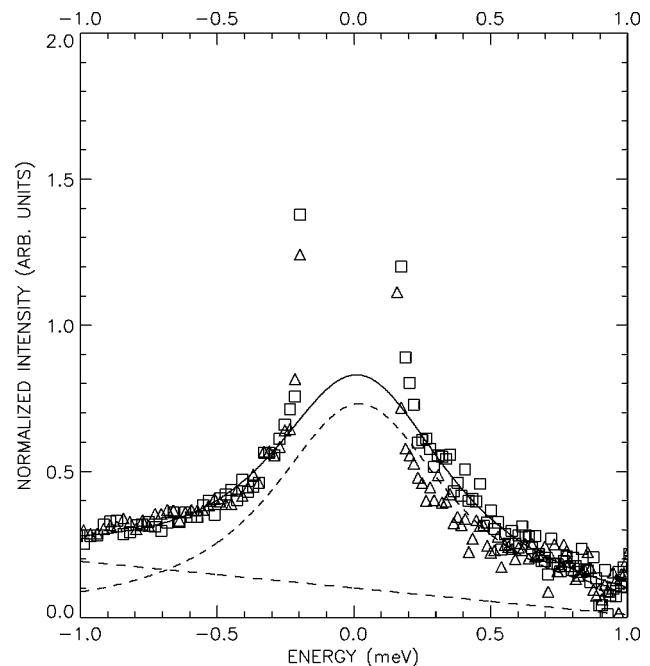


FIG. 7. Comparison of the data obtained with the  $\text{Ni}^{58}$  sample (squares) and the  $\text{Ni}^{60}$  sample (triangles).  $\lambda=5$  Å data. The data represent a sum over all detectors that yield quasielastic intensity. The intensity from the  $\text{Ni}^{60}$  sample has been multiplied by  $c=1.2$  and shifted by a constant  $7.5 \times 10^{-2}$ . The constant accounts for the change in the phonon density of states. Especially on the left hand side the data match perfectly. The small difference on the right hand wing might be due to a small contribution from a Ni-tagged phonon density of states. If Ni were responsible for the jump signal then there should have been a 25-fold increase on going from  $\text{Ni}^{60}$  to  $\text{Ni}^{58}$ .

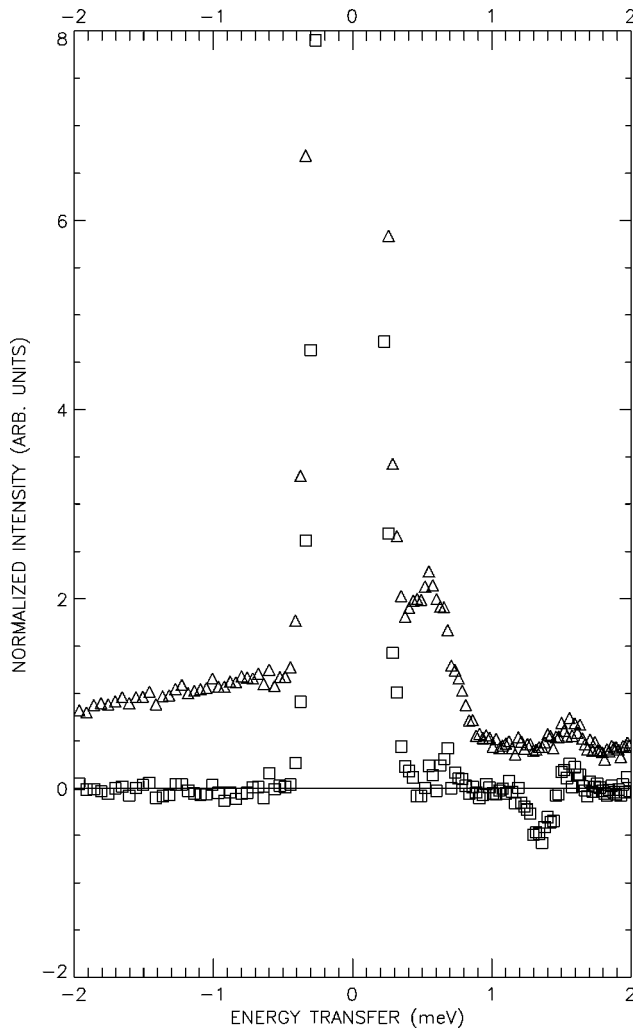


FIG. 8. Subtraction of the intensities from the two isotopic samples [ $\mathcal{I}(\text{Ni}^{58}) - 1.2 \times \mathcal{I}(\text{Ni}^{60})$ ] in the  $\lambda = 4 \text{ \AA}$  data at  $940 \text{ }^\circ\text{C}$  (squares). The data are compared with the unsubtracted  $\text{Ni}^{58}$  signal (triangles). Also this jump signal (at  $Q = 2.5 \text{ \AA}^{-1}$ ) does not imply the Ni atoms.

computations.<sup>25</sup> It can be expected that the TM ordering never did reach the equilibrium state during the experiment. Since it takes place by diffusion the ordering should be much more sluggish than the geometrical rearrangement. The large

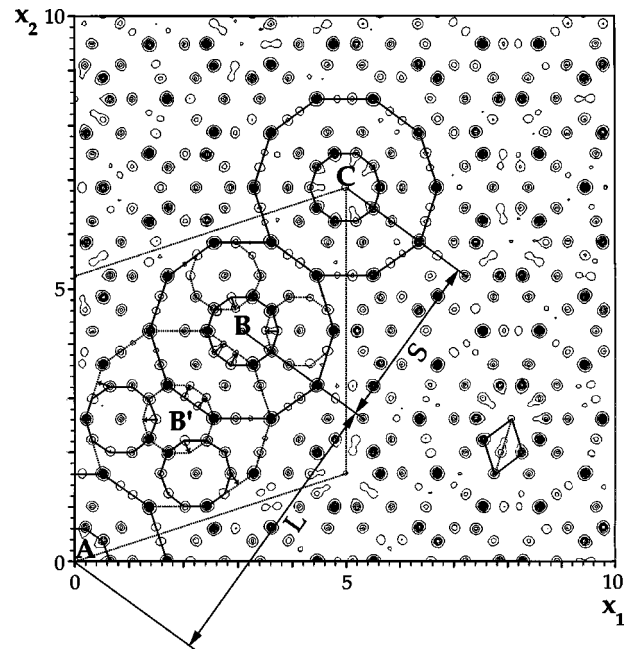


FIG. 9. Projected electron density map in relative units ( $37.94 \text{ \AA} \times 37.94 \text{ \AA}$ ) of decagonal Al-Co-Ni obtained from an x-ray structure analysis (Ref. 26). The columnar clusters (marked by A, B, C) with a sequence of distances  $LS$  are indicated by full lines ( $L = 19.79 \text{ \AA}$ ,  $S = L/\tau$ ). The flip  $LS \leftrightarrow SL$ , transporting the central cluster to the position indicated by dotted lines (marked by B'), is performed by  $0.938 \text{ \AA}$  jumps of a few Al atoms (marked by arrows between the split positions).

amount of diffuse scattering observed in *in situ* x-ray diffraction experiments bears out this assumption.<sup>21</sup>

#### IV. CONCLUSION

In conclusion, we have characterized atomic hopping in a decagonal phase. The general behavior of the jumps is very comparable to that observed in the icosahedral compounds. They also fulfill expectations founded on structural information.

#### ACKNOWLEDGMENTS

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