Plastic Deformation of Fine-Grained Al–Cu–Fe–(B) Icosahedral Poly-Quasicrystals at Elevated Temperature *

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Fine-grained Al–Cu–Fe–(B) icosahedral poly-quasicrystals (IQCs) as the main materials and fine-grained Al–Pd–Mn IQCs as the supplements, both prepared by powder metallurgy, are uniaxially deformed at various temperatures and strain rates. The systematic study shows the dependences of curves of the true stress versus true strain on several parameters, such as temperature, strain rate and grain size. For Al–Cu–Fe IQCs with grain sizes of about 10–30 μm, QC-specific intra-granular softening drop appears in the deformation curves at lower temperatures and/or faster strain rates, but disappears in those curves at higher temperatures and/or slower strain rates, which suggests that the inter-granular effects such as grain-boundary sliding should be taken into account to interpret the continuous hardening, similarly to conventional poly-crystals. For Al–Cu–Fe-B IQCs with smaller grain sizes of about 1 μm and fine-grained Al–Pd–Mn IQCs with grain sizes of about 10 μm, QC-specific intra-granular softening drop is absent for all the deformation curves at the possible lowest temperature and fastest strain rate. This implies that the smaller the grain size, the more the inter-granular contribution. At the same time, due to the rapid recovery caused by intense diffusion in small-sized grains, the intra-granular quasicrystal lattice reorders rapidly from disordering, which also inhibits the intra-granular softening drop to some extent.

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The mechanical properties and related microscopic mechanism of single-grained Al–Pd–Mn icosahedral quasicrystals (IQCs) have been understood much better than those of other kinds of IQCs, due to the successful growth of single-grained samples.[1−8] Macroscopically, as the main behaviour, all the curves of true stress versus true strain show the obvious intra-granular QC-specific softening drop beyond the maximum stress, similar to the curve shown in Fig. 1(a).

The macroscopic QC-specific softening drop in the curves of true stress vs true strain is ascribed to the intra-granular phasonic disordering left by moving dislocation. It is now accepted that the dislocation motion is a predominant intra-granular process mediating the high-temperature plastic deformation of single-grained Al–Pd–Mn IQCs although several groups are still debating upon gliding[1−3] or climbing,[4] working softening[5,6] or disordering softening.[1−3,5] The scenario of dislocation motion in single-grained Al–Pd–Mn IQC during the elevated temperature plastic deformation may be briefly expressed as follows. The phonon component of a dislocation on a densely packed plane of a QC may climb and/or glide under applied stress leaving a phasonic stacking fault behind. Owing to the strong intermetallic bonds in clusters in a QC and also large stacking fault energies, the critical stress for dislocation movement is very high. Hence almost all the QCs are brittle at room temperature. At elevated temperature, however, thermal activation helps to move dislocations, which circumvent or cut these clusters in the course of plastic deformation. The phasonic stacking fault left by a moving dislocation, which softens the QC matrix, will broaden and then disappear due to atomic diffusion at high temperature. Then, the partial dislocation becomes a perfect dislocation and the IQC structure is recovered. Both the phason-type disordering (or structure damage) induced by the motion of dislocation and the structure reordering (or structure recovery) due to atomic diffusion play important roles in the deformation process.

Nowadays, Al–Cu–Fe poly-grained IQCs may be the most extensively practically utilized QC materials, for example, coating. However, unlike the mostly studied single-grained Al–Pd–Mn IQC sample, highly perfect Al–Cu–Fe single-grained IQC is hardly produced, which to some extent inhibits the understanding of the macroscopic plastic properties and corresponding microscopic mechanism.[8−20] Apparently, the situation for Al–Cu–Fe poly-grained IQC is more complicated. Although some authors point out that dislocation motion may also accommodate the strain to some extent,[8,15−20] other intra-granular mechanisms, such as phase transformation and formation of voids,[9] phason-controlled atomic

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diffusion,[10] deformation twinning,[11,12] martensitic-type transformation,[13] are also proposed. Besides the above-mentioned intra-granular mechanisms, the grain boundary sliding is also argued as a factor in the deformation process of the Al–Cu–Fe poly-grained IQC.[14−16] Macroscopically, some authors argue that there may be two or more regimes, where different microscopic processes may be applied.[10,14−17] Therefore, it is necessary to obtain systematic mechanical information of Al–Cu–Fe poly-grained IQCs at elevated temperature.

In traditional poly-grained crystals, the strain may be accommodated by two main parts: the intra-granular part ε_G and the inter-granular ε_J. The proportion of ε_J will increase with decreasing grain size and strain rate, or increasing temperature.[21] Therefore, the inter-granular effect on the deformation of Al–Cu–Fe poly-grained IQC should be taken into consideration.

We perform mechanical testing at various temperatures and strain rates, on fine-grained Al–Cu–Fe–(B) IQCs as main studied materials and fine-grained Al–Pd–Mn IQCs as supplemented materials, which are prepared by powder metallurgy. From the systematic experimental results, we can differentiate the intra-granular and inter-granular effects and draw some preliminary qualitative conclusions on the role of temperature, strain rate, and grain size in the process of plastic deformation of the fine-grained IQCs roughly by comparing the shape of the true stress vs true strain curves. Detailed data fitting and the corresponding TEM microscopic mechanism study will be given later due to the length limit of this Letter.

Powder metallurgy is a very useful technique to produce single-phased poly-grained alloys, which are hardly produced by conventional methods. $Al_{65.1\,at\%}Cu_{22.6\,at\%}Fe_{12.1\,at\%}$ (grain sizes of about 10–30 μm, mainly around 10 μm, referring to Al–Cu–Fe) and $Al_{70.5\,at\%}Pd_{21\,at\%}Mn_{8.5at\%}$ (most grain size of about 10 μm, referring to Al–Pd–Mn) single-phased poly-grained IQCs produced by hot isostatic pressing (for producing method and characterization to see Ref. [22]), nominal composition $Al_{59\,at\%}Cu_{25.5\,at\%}Fe_{12.5\,at\%}B_{3\,at\%}$ (grain size of about 1 μm, referring to Al–Cu–Fe–B) single-phased poly-grained IQC produced by low uni-axial pressing (producing method similar to Ref. [23]) are used in the testing. Al–Cu–Fe-(B) samples are the main research objects and the Al–Pd–Mn samples are the supplements used to verify our argument.

The $2 \times 2 \times 5\,mm^3$ sized cuboid samples for mechanical testing are carefully produced by mechanical cutting, lapping, and polishing. Then, the samples are uni-axial-deformed by compression along the long axis on a Zwick Z050 testing system. The systematic true stress vs true strain curves (referring briefly to curves hereafter) are obtained at various temperatures and strain rates. In order to obtain more thermodynamic deformation parameters, several testing techniques including stress relaxation, temperature changing, and strain rate changing are utilized in each curve, which makes the curve complicated.

Figure 1(a) shows a typical curve of true stress σ vs true strain ε[21,24−26] for Al–Cu–Fe fine-grained IQC mainly at 600°C with strain rate $10^{-4}s^{-1}$, in which R_n means the n-th stress relaxation, TC_n for the n-th temperature changing, and SRC_n for the n-th strain rate changing. Unloading and reloading samples are necessary when TCs are applied during deformation. For better understanding of the main deformation feature of the curve, dotted lines are drawn for guide of eyes. The similar characteristics in the curves shown in other figures have the same meanings and no more dotted lines are given to avoid complicating the curves. Figure 1(a) clearly shows the QC-specific disordering softening drop beyond the maximum stress, which implies that the intra-granular effect predominately applies at the temperature and strain rate. It is noticed that the fine-grained IQCs show excellent plasticity (strain up to 16–20% without cracking), which also indicates the high quality of our samples produced by powder metallurgy.

From the information provided by the above techniques, we can obtain the macroscopic thermodynamic deformation parameters like activation volume V^* and activation enthalpy, etc. The activation volume can be obtained by stress relaxation here of about $1.34\,nm^3$ at the initial stress 218 MPa, which is comparable to the results for single-grained Al–Pd–Mn and poly-grained Al–Cu–Fe IQCs provided in literature. Another technique for obtaining V^* is strain rate change (SRC). From the above techniques we can give the $V^* \sim \sigma$ curve as shown in Fig. 1(b), which clearly shows that V^* decreases with increasing σ. More detailed treatment will be given elsewhere. We will focus on the main qualitative trend of the $\sigma \sim \varepsilon$ curves as mentioned above.

In order to know the dependence of the curves on temperature, the $\sigma \sim \varepsilon$ curves of Al–Cu–Fe fine-grained IQCs for various temperatures at the same strain rate of $10^{-4}s^{-1}$ are obtained, as shown as Fig. 2. By comparing the curves, we notice that the intra-granular QC-specific disordering softening drop predominates at relatively lower temperature, for example, 500°C. When we increase the temperature, the intra-granular disordering drop decreases gradually. When the temperature is at or above 650°C, we can only observe the continuous strain hardening without softening drop, implying that the intra-granular effect is suppressed in deformation at higher temperature.

To show the dependence of the mechanical behaviour on strain rate, the $\sigma \sim \varepsilon$ curves of Al–Cu–Fe fine-grained IQCs for various strain rates at the same temperature of 650°C are shown in Fig. 3. From the curves, we can find that under the higher temperature 650°C, the intra-granular QC-specific disorder-

ing drop shows again when the strain rate is increased from $10^{-4}\mathrm{s}^{-1}$ to $3\times10^{-4}\,\mathrm{s}^{-1}$. This means that faster strain rate is beneficial to observing intra-granular disorder softening effect.

According to the statement in the introduction, we propose preliminarily that: (1) similar to the traditional poly-grained crystals, the inter-granular strain dominates the deformation process under higher temperature and/or slower strain rate, under which the intra-granular effect such as QC-specific softening is not obvious; (2) the intense diffusion in the fine-grained Al–Cu–Fe IQCs reorders the phasonic disordering left by dislocation moving, which also inhibits the intra-granular softening effect to some extent. Therefore, we can only obtain the predominately QC-specific intra-granular softening effect by increasing the strain rate and/or decreasing the temperature, which can depress the inter-granular effect and/or the intra-granular diffusion effect efficiently.

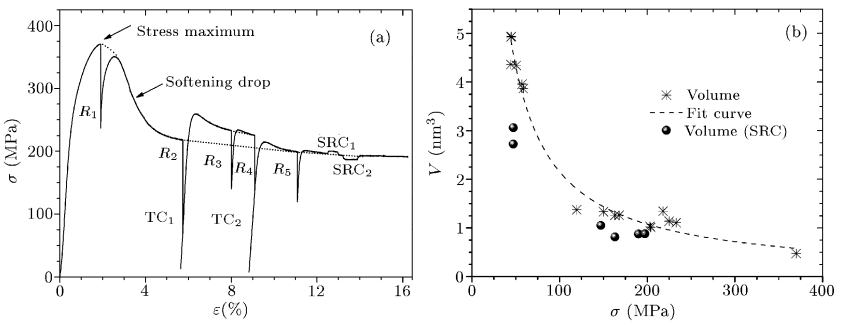


Fig. 1. (a) True stress σ of the Al–Cu–Fe fine-grained IQC vs true strain ε under 600°C and with strain rate $10^{-4}\mathrm{s}^{-1}$; (b) relationship between activation volume V^* and true stress σ.

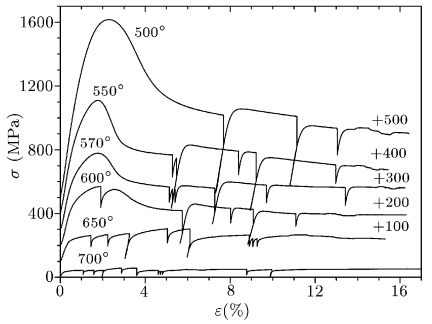


Fig. 2. True stress of the Al–Cu–Fe fine-grained IQC vs true strain under various temperatures and with the same strain rate $10^{-4}\mathrm{s}^{-1}$. Here the numbers on the right have been added to the real stress for clarity.

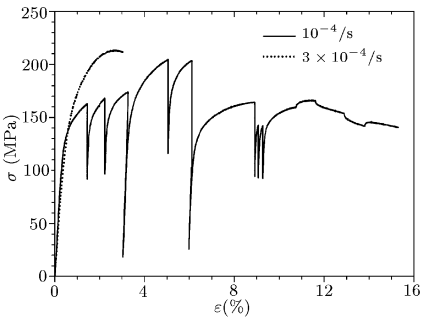


Fig. 3. True stress of the Al–Cu–Fe fine-grained IQC vs true strain under the same temperature 650°C and with various strain rates.

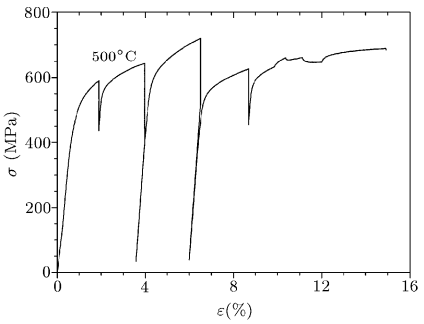


Fig. 4. True stress of the Al–Cu–Fe-B fine-grained IQC vs true strain under the temperature 500°C and with strain rate $10^{-4}\mathrm{s}^{-1}$.

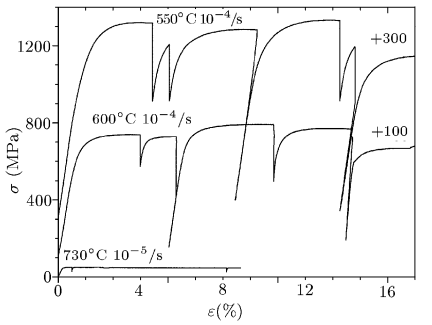


Fig. 5. True stress of the Al–Pd–Mn fine-grained IQCs vs true strain under various temperatures and with various strain rates. Here the numbers on the right have been added to the real stress for clarity.

Furthermore, it is expected that the above two arguments will be more obvious when the grain size of the IQC is smaller. To confirm these arguments, we test the Al–Cu–Fe-B (grain size much smaller) single-phased fine-grained IQC in the same way. Due to more intense diffusion in the finer-grained sample and maybe more grain boundary gliding, the intra-granular QC-specific disorder softening phenomenon cannot be observed even at temperature down to 500°C and with strain rate of $10^{-4}\mathrm{s}^{-1}$, as shown in Fig. 4. We can compare it with the curve of Al–Cu–Fe at the same temperature and strain rate shown in Fig. 2, where the intra-granular disordering drop is obvious. The strain is accommodated by the inter-granular effect, such as grain boundary sliding, which is serious in the finer-grained sample. More than that, the intra-granular reordering enhanced by intense diffusion may also depress the softening more in the finer-grained samples.

In other words, if we regard the grain size for single-grained IQC as infinite, we can expect the intra-granular softening effect at much higher temperature and much slower strain rate, which is exactly the case. The softening effect is obviously observed in Al–Cu–Fe single-grained IQC even at the temperature as high as 760°C and the much slower strain rate of $10^{-5}\mathrm{s}^{-1}$.[27] We can also observe the same trend in the $\sigma\sim\varepsilon$ curves caused by the grain size effect when we compare the

data here with those in the literature,[10,14–19] where the average grain sizes are larger than those of our Al–Cu–Fe samples and much larger than those of our Al–Cu–Fe-B ones.

The same effect can be extended into the Al–Pd–Mn IQCs system. As we know, the intra-granular softening effect can be obviously observed in the single-grained Al–Pd–Mn IQCs. However, as shown in Fig. 5, the Al–Pd–Mn single-phased poly-grained IQCs do not show the intra-granular softening drop evidence in the curves even when we increase the strain rate from $10^{-5}s^{-1}$ (mostly used in the testing on single-grained Al–Pd–Mn IQCs[1–3]) to $10^{-4}s^{-1}$ and decrease the temperature from 730°C (mostly used in the testing on single-grained Al–Pd–Mn IQCs[1–3]) to 550°C. All the curves show obvious continuous strain hardening, which indicates that the intra-granular disordering softening drop effect is obviously suppressed. The sample is broken when it is tested at 500°C. We can not try lower temperature and faster strain rate, which is dangerous to both the testing system and the tested sample. It also verifies our arguments.

Therefore, the inter-granular mechanism in the poly-grained, especially fine-grained IQCs should be considered in the plastic deformation process, and the corresponding intra-granular QC lattice reordering due to higher temperature, slower strain rate, and smaller grain size also inhibits the softening effect to some extent. Thus, we should use the corresponding mechanical testing conditions to show more inter-granular or intra-granular contribution. This may agree with the argument about two or more regimes for the mechanical properties in the literature.[10,14–17] Microscopic mechanism study aided by TEM should also be taken on different deformed samples to differentiate the inter-granular and intra-granular contributions.

In conclusion, we have performed systematic mechanical testing at various temperatures, with various strain rates, on fine-grained Al–Cu–Fe-(B) poly-IQCs, which are prepared by powder metallurgy. From the experimental results, we can draw the preliminary qualitative conclusion on the role of grain size in the process of plastic deformation of the poly-grained IQCs roughly. Similar to traditional poly-crystals, the inter-granular strain dominates the deformation under higher temperature, slower strain rate, with smaller grain size sample, where the true stress vs true strain curves show continuous hardening without the QC-specific softening drop. At the same time, due to the rapid recovery caused by intense diffusion in small-sized grains, the intra-granular quasicrystal lattice reorders rapidly from disordering, which inhibits the disordering softening effect to some extent. It is obvious that grain size plays a very important role in the plastic deformation of the fine-grained IQCs. Further data treatment and microscopic mechanism analysis are in progress.

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