PACS: 61.50.Ks, 62.20.-x

Ye. Teytel

STRUCTURAL ASPECTS OF SEVERE PLASTIC DEFORMATION

PhD USA, semiretired

Received January 10, 2019

The analysis of the structure and phase composition of alloys processed by severe plastic deformation (SPD) has been carried out. The data about the effect of the processing parameters on the results of low-temperature dynamic recrystallization are reported.

Keywords: point defects, severe plastic deformation, equivalent temperature, quasiheating, superplasticity, amorphization, low temperature dynamic recrystallization, super fragmentation

1. Introduction

There are five most common methods which can provide severe plastic deformation: rotation under pressure (RP), mechanical milling and grinding, mechanical alloying, twist extrusion and equal channel angular pressing. The most interesting and looking like strange results were obtained for the first three methods.

In [1], the authors stated that the structures formed upon SPD were very unusual and unpredictable. However, in [2] it was shown that upon SPD alloys achieved so high density of defects (that was energy), that thermodynamic parameters of materials were significantly changed; practically, alloys moved to a higher temperature on phase diagrams (PDs). Earlier, in 1984, G. Martin published a similar idea for irradiation, demonstrated application for PDs, and used a new term, namely, the effective temperature of material $T_{\rm eff}$ [3]. However the present author thinks that the term «equivalent temperature» $T_{\rm eq}$ (to the standard temperature) is more correct (see below). Much later, in 1995, H. Bakker [4] used this conception for the analysis of phase transformations upon mechanical grinding. The problem of detailed explanation is often the lack of having particular PDs obtained under the used hydrostatic component of pressure.

Unfortunately, these works still have not found an appropriate attention of world scientists. Nevertheless, we showed in [5,6], how fruitful this conception was. In [5] we also introduced a new term (quasiheating) and applied T_{eq} to other types of intensive impacts, such as accelerated diffusion causing amorphization in thin films, etc.

Since after the termination of deformation, no significant changes in the dislocation structure are observed (usually there is only a small loss of point defects), the main difference between moving and standing dislocations is that the first ones generate point defects after intersections with others and creation of steps on dislocation lines. It means that the alloy's behavior depends significantly on the moving defect density. Probably, huge density of point defects is generated in the microvolumes, where the density of defects achieves the critical value due to rotation and non-crystallographic motion of dislocations that causes formation of different structures and phase transformations, including amorphization [7,8].

Because of heterogeneous nature of plastic deformation, in real samples, amorphization and other deformation-induced transformations go gradually with the deformation degree rather than with a jump, as it probably is in micro volumes. Similar behavior was found, for instance, for atomic disorder drawing [9].

In [10], nickel is rolled to 60%, exhibited partial amorphization and has the dislocation density of 10^{13} – 10^{14} . The elastic energy for the last value is calculated as 2.2 kJ/mol, that is significantly lower than the melting one equal to 3.3 kJ/mol. This means that 1.1 kJ/mol is associated with point defects.

Equilibrium concentration of vacancies in metals at room temperature is around 10^{-18} and, being estimated by different methods, this value can achieve ~ 10^{-4} under SPD [7,8] and probably it is higher at temperatures lower than 273 K. That are typical pre-melting numbers and this fact greatly increases the rate of structure and phase transformations and causes unusual mechanical properties of alloys [11]. But T_{eq} can exceed T_{melt} and the thermal decomposition/vaporizing (sublimation) phase transformation numbers that can be concluded from the fact that some oxides become decomposed or exploded upon RP [12].

The data on amorphization due to irradiation in thin films give evidences that it is sufficient to have only point defects in large numbers for this transition. In [2], it was shown that in the process of RP of MnAl–C, low temperature dynamic fragmentation proceeded fully very quickly (in ~ 2 min). The process occurred practically instantly in slip bands that are in the course of their formation. Similar situation was found for amorphization in the samples deformed at the liquid-nitrogen boiling point (TLN).

In [13], the diffusion coefficient upon RP was calculated by using the time of dissolving of copper particles in cobalt matrix and it was found that the diffusion rate of Cu increased by 22 orders!

Now we should say some words about mechanical alloying. It can be used mainly for:

1. Production of alloy powders without melting.

2. Increase of solubility of some elements. An example is reported in [14] where the authors managed to increase solubility of Fe in Al from 0.03 up to 2.2 wt% and gained microhardness increase from 750 up to 2550 MPa.

3. Obtaining of nano- or amorphous structure or sometimes quasicrystals. According to design of most PDs, it is possible to get a solubility increase, but in some cases, like Mg + V, these elements do not mixture in the liquid state up to $3000^{\circ}C$, so no alloying effects can be expected. 4. Synthesis of alloys, chemicals and intermetallics.

Some time ago an unexplained fact was reported in [15]. The authors used mechanical alloying for obtaining vanadium carbide VC from powders V and C. They said it was possible only if the maximum power of machines was used. At a lower energy, they got nano-vanadium and amorphous carbon (both existed on PD). Here is a probable explanation. If the growing energy of the generated defects (the sum energy of them) is not big enough to achieve the melting energy quickly, it is necessary to transfer the material to amorphous state with required compositions (in this case VC). Formation of other phases with a lower energy according to the real (usually changed) PD is possible (the diffusion rate is huge). Annealing might not practically return material to the initial state for reasonable time because the diffusion rate in this case will be low (standard). This is not a symmetric situation.

An interesting question could be asked: in what case we have mechanical alloying phenomenon, and when the opposite process of decomposition of chemicals and intermetallics takes place? The most probable answer is: when $T_{eq} > T_{boil}$ (thermal decomposition), we will have decomposition (the example Mn₂O₅ transfers to Mn₂O₃ + O₂ with explosion [12]) and the opposite case is related to mechanical alloying. To have so high T_{eq} , we should have a material with very high yield stress and strengthening coefficient and high boiling point.

In [16], intermetallic NiTi was milled for different time and strange results were found, namely, a significant difference with RP of MnAl–C, an unusual type of low temperature dynamic recrystallization (LTDR): small recrystallized grains were formed at the early stage of deformation (probably because of high energy of defects as a result of martensitic transformation) and then transferred to amorphous state. Their size was gradually reduced down to 5 nm (the critical number for amorphization, as evaluated in [16]). A possible explanation of this behavior was that many point defects probably disappeared during the pauses between strokes. Real time of one impulse of plastic deformation does not exceed 0.1 s that is probably not enough for creating conditions for standard LTDR and superplasticity deformation mechanism (see below). An additional research is needed to understand this situation.

Now we will consider the most possible types of structures after SPD (with some important comments).

1. As a result of LTDR, one or several recrystallized phases are formed. This case is typical of metals and industrial alloys. It is possible to explain a lot of literature data related to the behavior of the second phases such as dissolution of big particles and simultaneous precipitation of much smaller ones [7]. The reason is obviously minimized energy due to LTDR that transfers alloys to the mode of superplasticity of plastic deformation. Practically, creation of the appropriate grain size of each phase occurs. The chemical composition of phases and their quantities are expected to be changed in accordance with the actual PD and T_{eq} value.

2. Single fragmented phase is formed. In some alloys (for example, MnAl–C, RP, 20-C [2]), the fragments with the average diameter of \sim 50 nm are randomly

disoriented and not elongated, contrary to uniaxial deformation methods (like drawing, etc.). The process can be named super-fragmentation. An important fact is that the rotation number n dependence of the microhardness at $n \sim 3$ becomes close to saturation that means no other (further) transformation is possible. This fact is an evidence of the existence of a stable structure with the lowest energy. The mechanism of plastic deformation is similar to superplasticity involving probably migration of boundary fragments. If we increase deformation speed or decrease the temperature of SPD, we probably will obtain smaller and smaller fragment size, and the alloy will be expected to transfer to amorphous state at some virtual critical diameter of the fragments.

3. Single amorphous phase. An example is deformation of MnAl–C at low temperature (TLN) [2]. It is important that RP transfers the amorphous state obtained by SPD at TLN to the same fragmented one as room temperature deformation. This fact means that the transition of a crystal to amorphous state is reversible with respect to the deformation temperature and there is a critical temperature for this transformation. It probably depends on the deformation speed.

It is known that at lower temperatures, the strengthening is higher than at room temperature, that will cause T_{eq} to grow and the diffusion speed will accelerate. This phenomenon can be used for improvement of mechanical alloying speed (possibly patentable method). The slowest increase of T_{eq} is supposed to be for FCC pure metals like Cu, Ni, Al, etc. A possible application is as follows: if one needs to obtain maximally homogeneous material, he has to quasiheat the alloy to the single phase area (liquid is the better variant) and to obtain 100% of amorphous or other single-phase state (possibly patentable method).

4. Solid recrystallized crystalline phase and amorphous one means that the alloy is in the state with crystalline solid + liquid phases under the PD. If T_{eq} is close to liquidus, the structure will consist of amorphous phase surrounding small crystalline particles. It probably might cause a wrong impression that the crystal phase directly transfers to the amorphous state. Actually, the situation is more complex: the alloy should decompose into two phases of different composition (solid + liquid). In the case of alloy location at the line of eutectic composition, we can see the direct transformation of crystal phase to amorphous state.

In [17], the authors found what the structure caused the improvement of magnetic induction of the $Fe_{81}B_{11}Si_6$ alloy by 40% (!) by using RP. From the viewpoint of the described concept, this effect can be explained by the presence of two or more nano-phases and significant redistribution of iron between them (it can be also case 1). Obviously, this phenomenon can be reproduced in industry by using mechanical milling or alloying methods.

It is possible that in some situations, deformation temperature higher than the room one should be applied in order to avoid jumping over the needed structure area on PD. This method can be possibly used for making new types of catalysts.

5. Two amorphous phases found in [11] but not explained. It was possible because the alloy on PD was in two different liquids areas. 6. Structures reflecting cyclic transformation from AS to crystal phase and back were reported in several articles [18,19], some with different complex mathematical models of this phenomenon. We suggested a simple explanation [5] based on heat release and its consumption upon phase transformations. No structure information is available.

Here are the main definitions. It is reasonable to define SPD as a big ($e > \sim 1$) plastic deformation at the temperature in the vicinity of room one and lower, with more than one loading directions when diffusion controlled transformations such as super fragmentation, LTDR or amorphization take place

So there are three final stable (equilibrium) structures: fragmented one (MnAl–C, 20°C, RP), recrystallized (metals and alloys at 77–273 K) and amorphous one (some intermetallics: MnAl–C at 77 K and NiTi, ZrCo, NiZr₂ at 273K).

There is an important role of quasihydrostatic pressure that prevents rapture and cracking and results in reduced mobility of vacancies and their higher concentration.

Equivalent temperature T_{eq} is *T*-coordinate of alloy at which the corresponding PD obtained under quasihydrostatic pressure obeys all PD rules: type of phases, quantity of each phase and their chemical compositions. T_{eq} can be evaluated from the structure/phase analysis with using PDs (see [13]). For PDs, it does not matter what kind of energy is applied.

Quasyheating is a process of increasing the accumulated energy (practically the sum of the energies of all defects) by any type of plastic deformation, irradiation, diffusion in thin films, etc. It is reasonable to say, that if somebody virtually touches a sample upon SPD with, for instance, $T_{eq} = 1000^{\circ}$ C, he is expected to feel not so hot (only the standard heat).

Author gratefully acknowledge dr. V. Pilugin for introducing author in RP method and deform several samples, dr. A. Yermakov (he first discovered deformation induced amorphization) for help with information/literature.

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- Е. Тейтель

СТРУКТУРНЫЕ АСПЕКТЫ ИНТЕНСИВНОЙ ПЛАСТИЧЕСКОЙ ДЕФОРМАЦИИ

Проведен анализ структуры и фазового состава сплавов, прошедших обработку интенсивной пластической деформацией. Приведены данные о влиянии параметров обработки на результаты низкотемпературной динамической рекристаллизации

Ключевые слова: точечные дефекты, интенсивная пластическая деформация, эквивалентная температура, квазинагрев, суперпластичность, аморфизация, низкотемпературная динамическая рекристаллизация, суперфрагментация