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Professor Harry Bhadeshia FRS is head of the Phase Transformations and Complex Properties Research Group at the University of Cambridge. He works on the theory of solid-state transformations with a view to attempting the design of metallic alloys, particularly those based on iron. He is the author or co-author of several textbooks and many research papers and review articles, the details of which can be found at <http://www.msm.cam.ac.uk/phase-trans>

The RMS was extremely fortunate to have Professor Harry Bhadeshia to deliver this year's Annual Materials Lecture on the topic of Mechanically Alloyed Metals. To a full auditorium in St John's College, Oxford, Professor Bhadeshia described the process by which these fascinating materials are made and the extraordinary properties they can attain. He showed some stunning microstructures: grains that are nanometres in size; others that are metres in length; and grains that twist, turn and convolute in three dimensions. He then took us on a tour of thermodynamics to indicate how these materials can show us the fundamentals of solution processes and the nucleation of grain recrystallisation. It was a very interesting and stimulating presentation, expertly and enthusiastically given, which generated much debate. We all left for the Microscopy of Composites V dinner talking excitedly of mechanical alloying and the number of apples it would take to reach the moon.



Mechanical alloying involves the severe deformation of mixtures of powders until they form the most intimate of atomic solutions. Inert oxides can also be introduced to form a uniform dispersion of fine particles that strengthen the consolidated product. Large quantities of iron and nickel-base alloys with unusual properties are produced commercially using this process. The theory describing the way in which the powders evolve into a solution is reviewed. There are some fundamental constraints which dictate how the microstructure must change during mechanical alloying for the process to be at all viable. The strange recrystallisation behaviour of the alloys can be understood if it is assumed that unlike normal metals, the grains in the mechanically alloyed sample are not topologically independent.

Introduction

An alloy can be created without melting, by violently deforming mixtures of different powders; Figure 1 (Benjamin, 1970; Benjamin & Gilman, 1983; Cessinger, 1984; Hack, 1984). Inert oxides can, using this technique, be introduced uniformly into the microstructure. The dispersion-strengthened alloyed powders are then consolidated using hot-isostatic pressing and extrusion, to produce a solid with a very fine grain structure. Heat treatment then induces recrystallisation, either into a coarse columnar grain structure or into a fine, equiaxed set of grains. Columnar grains occur for two reasons: the oxide particles tend to become aligned along the extrusion direction, making that a favoured growth direction. Alternatively, and in the absence of particle alignment, columnar growth can be stimulated by recrystallising in a temperature gradient; the latter may be a stationary gradient or one which moves along the sample, as in zone annealing. The columnar microstructure is desirable in applications where the resistance to creep deformation is paramount.

The chemical compositions of some of the commercial alloys produced using this method are listed in Table 1. They all contain chromium and/or aluminium for corrosion and oxidation resistance, and yttrium or titanium oxides for creep strength. Yttrium oxide cannot be introduced into either iron or nickel by any method other than mechanical alloying; indeed, this was the motivation for the original work by Benjamin (1970).

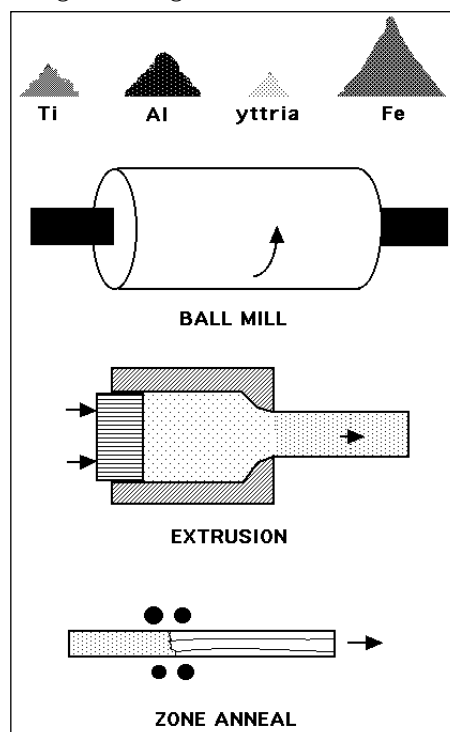


Fig. 1. The manufacture of mechanically alloyed metals for engineering applications. The elemental powders/master-alloys/oxides are milled together to produce solid solutions with uniform dispersions of oxide particles. This powder is consolidated and the resulting material heat-treated to achieve a coarse, directional grain structure.

Fe-base	C	Cr	Al	Mo	Ti	N	Ti ₂ O ₃	Y ₂ O ₃	Fe	
MA957	0.01	14.0	–	0.3	1.0	0.012	–	0.27	Balance	
DT2203Y05		13.0	–	1.5	2.2		–	0.5	Balance	
ODM 331		13.0	3.0	1.5	0.6		–	0.5	Balance	
ODM 751		16.5	4.5	1.5	0.6		–	0.5	Balance	
ODM 061		20.0	6.0	1.5	0.6		–	0.5	Balance	
MA956	0.01	20.0	4.5	–	0.5	0.045	–	0.50	Balance	
PM2000	<0.04	20.0	5.5		0.5		–	0.5	Balance	
PM2010	<0.04	20.0	5.5		0.5		–	1.0	Balance	
DT		13.0	–	1.5	2.9		1.8	–	Balance	
DY		13.0	–	1.5	2.2		0.9	0.5	Balance	
Ni-Base	C	Cr	Al	Ti	W	Fe	N	Total O	Y ₂ O ₃	Ni
MA6000	0.06	15.0	4.5	2.3	3.9	1.5	0.2	0.57	1.1	Balance
MA760	0.06	19.5	6.0	–	3.4	1.2	0.3	0.6	1.0	Balance
MA758†	0.05	30.0	0.3	–	0.5	–	–	0.37	0.6	Balance
PM1000†		20.0	0.3	0.5		3.0			0.6	Balance

Table 1. Compositions (wt%) of some typical alloys.

† MA758 and PM1000 are nickel base mechanical alloys without γ' strengthening. The compositions of ODM061, DT and DY are from Regele (1994), as are the nitrogen data for MA956 and MA957. The compositions of PM2000 and PM2010 are from Krautwasser *et al.* (1994).

Microstructure

Immediately after the mechanical alloying process, the powders have a grain size which can be as fine as 1-2 nm locally (Jaeger & Jones, 1991). This is hardly surprising given the extent of the deformation during mechanical alloying, with true strains of the order of 9, equivalent to stretching a unit length by a factor of 8000. The consolidation process involves hot extrusion and rolling at temperatures of about 1000°C, which causes recrystallisation into a sub-micrometre grain size (Figure 2). It is known that during the course of consolidation, the material may dynamically recrystallise several times (Jaeger & Jones, 1991). It should be emphasised that the sub-micrometre grains illustrated in Figure 2 are not low-misorientation cell structures, but true grains with large relative misorientations (Bhadeshia, 1997). Subsequent heat-treatment leads to primary recrystallisation into a very coarse grained structure whose dimensions may be comparable to those of the sample (Figure 2b).

The grains in Figure 2a are elongated because the hot-rolling leaves a microstructure with a residual deformation, with a dislocation density of about 10^{15} m^{-2} (Little *et al.*, 1991); although this is large, it is not particularly high when compared with dislocation densities found in conventional steel martensitic microstructures (Bhadeshia, 1998). The vast majority of the stored

energy of about 55 J mol^{-1} in the material is due to the very fine grain size ((Bhadeshia, 1997).

Chemical structure

The intense deformation associated with mechanical alloying can force atoms into positions where they may not prefer to be at equilibrium. The atomic structure of solid solutions in commercially important metals formed

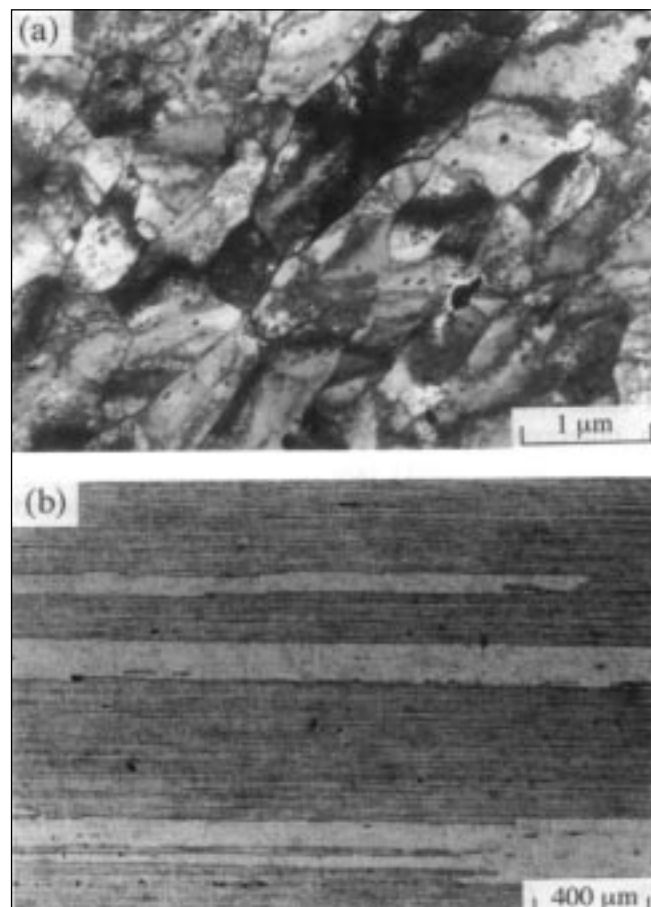


Fig. 2. (a) Transmission electron micrograph showing the sub-micrometre grain structure of mechanically alloyed and consolidated iron-base MA956 alloy. The micrograph is a section normal to the extrusion direction. (b) Optical micrograph showing the coarse, columnar recrystallisation grain structure resulting from heat treatment at temperatures as high as 1400°C.

by the mechanical alloying process has been studied using field ion microscopy and the atom-probe ((Chou *et al.*, 1993).

A solution which is homogeneous will nevertheless exhibit concentration differences of increasing magnitude as the size of the region which is chemically analysed decreases (Landau & Lifshitz, 1958; Russell, 1971). These are random fluctuations which obey the laws of stochastic processes, and represent the real distribution of atoms in the solution. These equilibrium variations cannot usually be observed directly because of the lack of spatial resolution and noise in the usual microanalytical techniques. The fluctuations only become apparent when the resolution of chemical analysis falls to less than about a thousand atoms block. The atom probe technique collects the experimental data on an atom by atom basis. The atom by atom data can be presented at any block size.

Figure 3 illustrates the variation in the iron and chromium concentrations in 50 atom blocks, of the ferrite in MA 956. There are real fluctuations but further analysis is needed to show whether they are beyond what is expected in homogeneous solutions.

For a random solution, the distribution of concentrations should be binomial since the fluctuations are random; any significant deviations from the binomial distribution would indicate either the clustering of like atoms or the ordering of unlike pairs.

The frequency distribution is obtained by plotting the total number of composition blocks with a given number of atoms of a specified element against the concentration. Figure 4 shows that the experimental distributions are essentially identical to the calculated binomial distributions, indicating that the solutions are random.

The atom probe data can be analysed further if it is assumed, fairly reasonably, that the successive atoms picked up by the mass spectrometer were near neighbour atoms in the sample. Successive atoms which are identical then represent bonds between like atoms etc. so that pair probabilities used in solid solution theory can be measured experimentally. These data can be compared against calculated pair probabilities. Thus, in a random A — B solution, the probability P_{AB} of finding A — B or B — A bonds is given by $P_{AB} = 2x_Ax_B$ where x_i is the atom fraction of element i . Similarly, $P_{AA} = x_A^2$

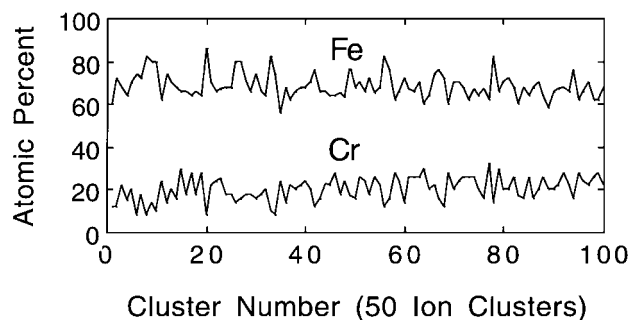


Fig. 3. The variation in the iron and chromium concentrations of 50 atom samples of MA956 (Chou *et al.*, 1993).

and $P_{BB} = x_B^2$. Table 2 shows the excellent agreement between the experimentally measured pair probabilities and those calculated assuming a random distribution of atoms.

This does not mean that the solutions are thermodynamically ideal, but rather that the alloy preparation method which involves intense deformation forces a random dispersal of atoms. Indeed, Fe-Cr solutions are known to deviate significantly from ideality, with a tendency for like atoms to cluster (Miller, 1988; Uemori *et al.*, 1988) Thus, it can be concluded that the alloy is in a mechanically homogenised nonequilibrium state, and that prolonged annealing at low temperatures should lead to, for example, the clustering of chromium atoms.

Solution formation

Normal thermodynamic theory for solutions begins with the mixing of component atoms. In mechanical alloying, however, the solution is prepared by first mixing together lumps of the components, each of which might contain many millions of identical atoms. We examine here the way in which a solution evolves from these large lumps into an intimate mixture of different kinds of atoms (Badmos & Bhadeshia, 1997). It will be shown later that this leads to interesting outcomes which have implications for how we interpret the mechanical alloying process.

Consider the pure components A and B with molar free energies μ_A^0 and μ_B^0 respectively. If the components are initially in the form of powders then the average free energy of such a mixture of powders is simply:

$$G\{\text{mixture}\} = (1 - x)\mu_A^0 + x\mu_B^0 \quad (1)$$

where x is the mole fraction of B. It is assumed that the powder particles are so large that the A and B atoms

Alloy	Element	P_{AA}	P_{AB}	P_{BB}	N
MA 956	Cr (Measured)	0.638	0.324	0.038	12168
MA 956	Cr (Calculated)	0.637	0.322	0.041	
MA 956	Al (Measured)	0.808	0.184	0.008	12168
MA 956	Al (Calculated)	0.833	0.160	0.008	

Table 2. Pair probability analysis. B is the solute element (such as Cr or Al) whereas A represents the remainder of atoms. N represents the total number of atoms included in the analysis. The calculations assume a random solution.

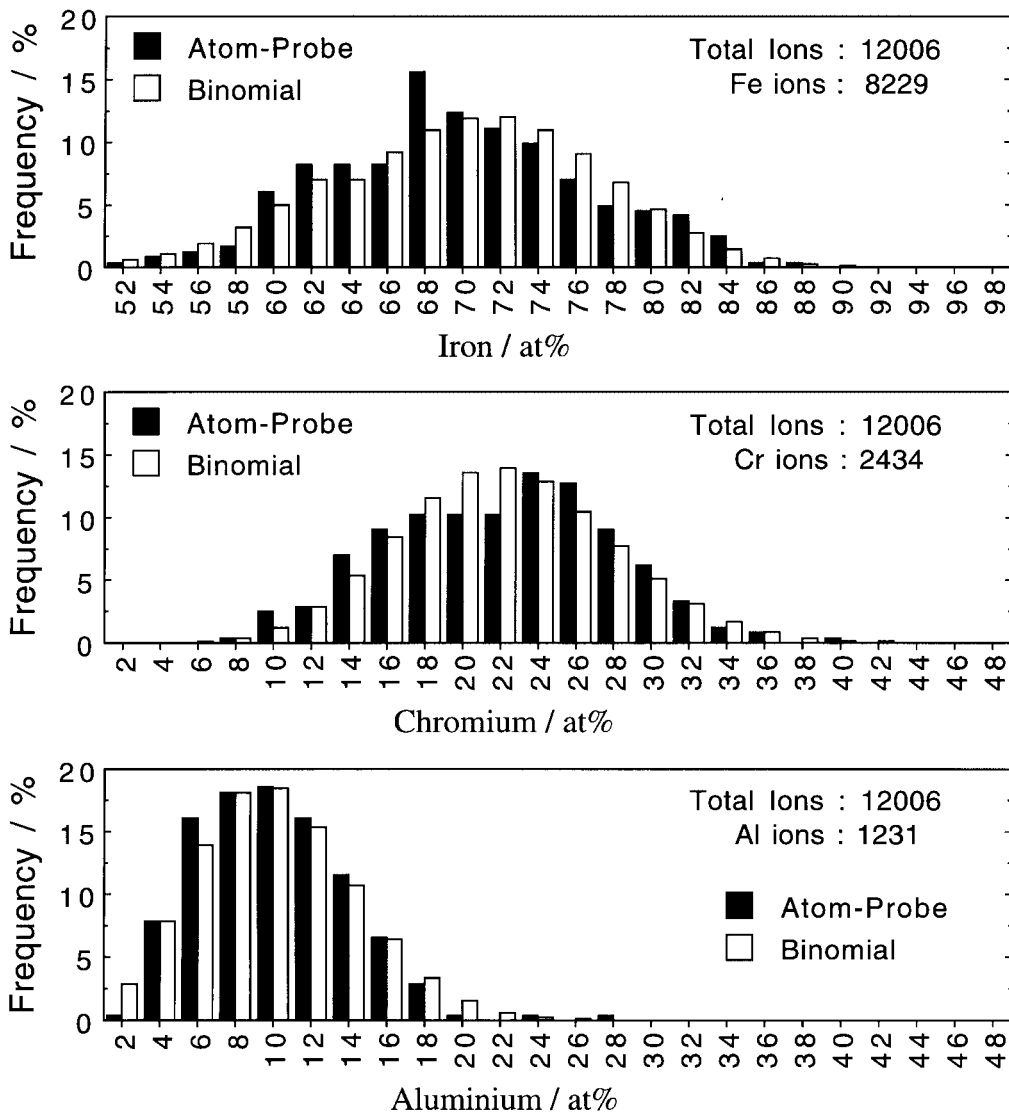


Fig. 4. Frequency distribution curves for iron, chromium and aluminium in mechanically alloyed MA956 (Chou *et al.*, 1993).

do not ‘feel’ each other’s presence via interatomic forces between unlike atoms. It is also assumed that the number of ways in which the mixture of powder particles can be arranged is not sufficiently different from unity to give a significant contribution to a configurational entropy of mixing. Thus, a blend of powders which obeys equation 1 is called a *mechanical mixture*. It has a free energy that is simply a weighted mean of the components, as illustrated in Figure 5a for a mean composition x .

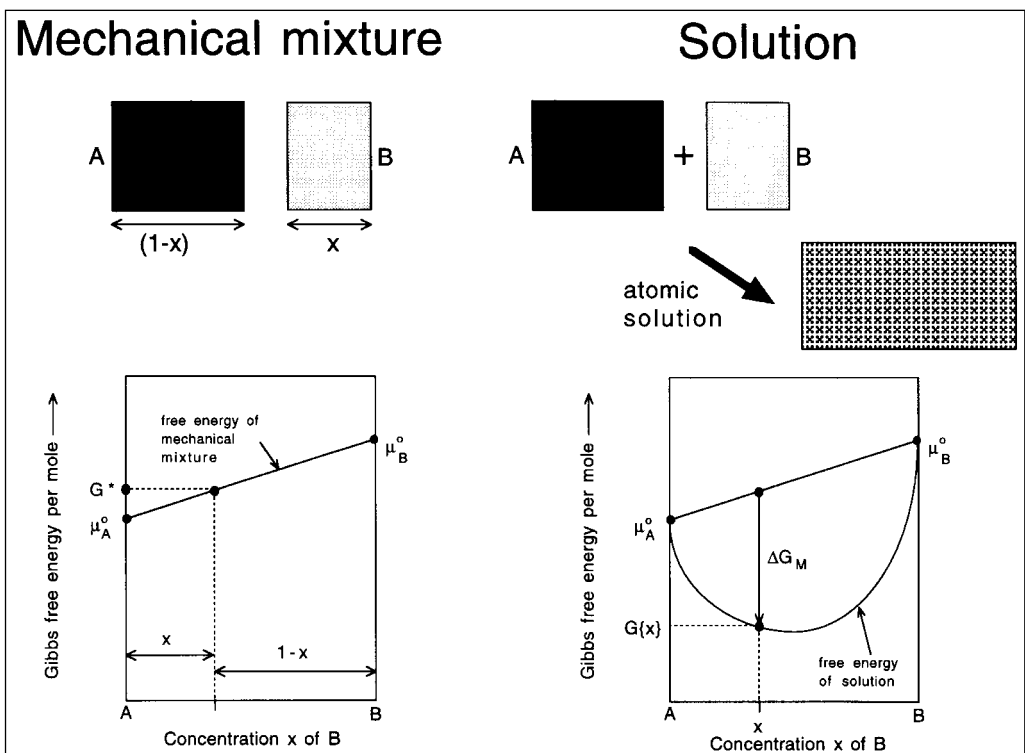


Fig. 5. (left) The free energy of a mechanical mixture, where the mean free energy is simply the weighted mean of the components. (right) The free energy of an ideal atomic solution is always lower than that of a mechanical mixture due to configurational entropy.

ventionally taken to describe a mixture of atoms or molecules. There will in general be an enthalpy change associated with the change in near neighbour bonds. Because the total number of ways in which the ‘particles’ can arrange is now very large, there will always be a significant contribution from the entropy of mixing, even when the enthalpy of mixing is zero. The free energy of the solution is therefore different from that of the mechanical mixture, as illustrated in Figure 5b. The difference in the free energy between these two states of the components is the free energy of mixing ΔG_M , the essential term in all thermodynamic models for solutions.

Whereas mechanical mixtures and atomic or molecular solutions are familiar in all of the natural sciences, the intermediate

In contrast to a mechanical mixture, a *solution* is con-

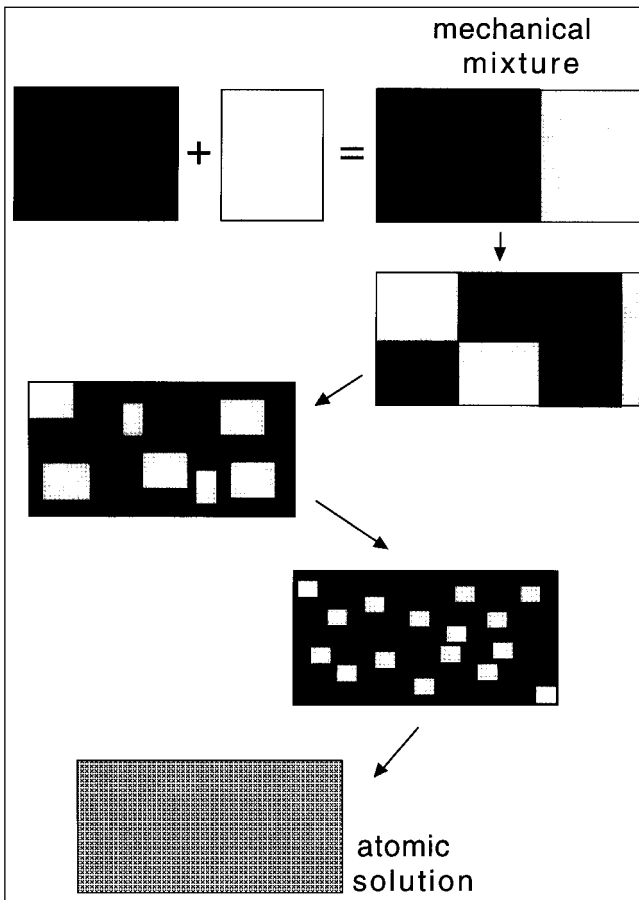


Fig. 6. Schematic illustration of the evolution of an atomic solution by the progressive reduction in the size of different particles, a process akin to mechanical alloying.

states have only recently been addressed (Badmos & Bhadeshia, 1997). The problem is illustrated in Figure 6, which shows the division of particles into ever smaller particles until an atomic solution is achieved. At what point in the size scale do these mixtures of particles begin to exhibit solution-like behaviour?

To answer this question we shall assume first that there is no enthalpy of mixing. The problem then reduces to one of finding the configurational entropy of mixtures of lumps as opposed to atoms. Suppose that there are m_A atoms per powder particle of A and m_B atoms per particle of B ; the powders are then mixed in a proportion which gives an average mole fraction x of B .

There is only one configuration when the heaps of pure powders are separate. When the powders are mixed at random, the number of possible configurations for a mole of atoms becomes:

$$\frac{(N_a([1-x]/m_A + x/m_B))!}{(N_a([1-x]/m_A))! (N_a x/m_B)!} \quad (2)$$

where N_a is Avogadro's number. The numerator in equation 2 is the total number of particles and the denominator the product of the factorials of the A and B particles respectively. Using the Boltzmann equation and Stirling's approximation, the molar entropy of mixing becomes (Badmos & Bhadeshia, 1997):

$$\begin{aligned} \frac{\Delta S_M}{kN_a} = & \frac{(1-x)m_B + xm_A}{m_A m_B} \ln \left\{ N_a \frac{(1-x)m_B + xm_A}{m_A m_B} \right\} \\ & - \frac{1-x}{m_A} \ln \left\{ \frac{N_a(1-x)}{m_A} \right\} \\ & - \frac{x}{m_B} \ln \left\{ \frac{N_a x}{m_B} \right\} \end{aligned} \quad (3)$$

subject to the condition that the number of particles remains integral and non-zero. As a check, it is easy to show that this equation reduces to the familiar

$$\Delta S_M = -kN_a[(1-x) \ln\{1-x\} + x \ln\{x\}]$$

when $m_A = m_B = 1$.

Naturally, the largest reduction in free energy occurs when the particle sizes are atomic. Figure 7 shows the molar free energy of mixing for a case where the average composition is equiatomic assuming that only configurational entropy contributes to the free energy of mixing. An equiatomic composition maximises configurational entropy. When it is considered that phase changes often occur at appreciable rates when the accompanying reduction in free energy is just 10 J mol^{-1} , Figure 7 shows that the entropy of mixing cannot be ignored when the particle size is less than a few hundreds of atoms. In commercial practice, powder metallurgically produced particles are typically $100 \mu\text{m}$ in size, in which case the entropy of mixing can be neglected entirely, though for the case illustrated, solution-like behaviour occurs when the particle size is about 10^2 atoms.

Enthalpy and interfacial energy

The enthalpy of mixing will not in general be zero as was assumed above. The binding energy is the change

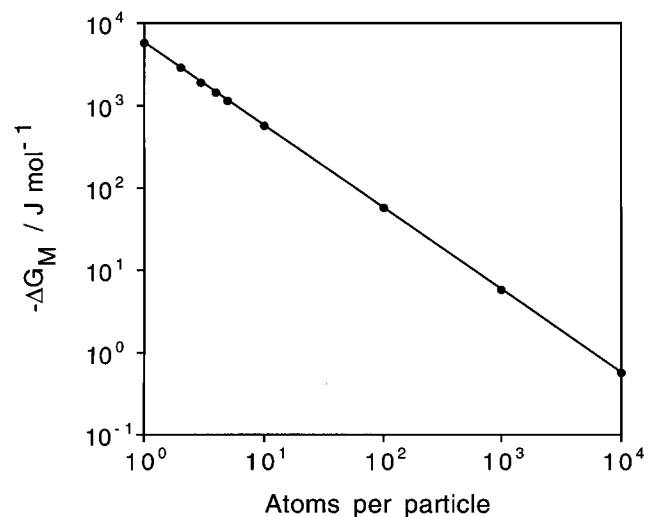


Fig. 7. The molar Gibbs free energy of mixing, $\Delta G_M = -T\Delta S_M$, for a binary alloy, as a function of the particle size when all the particles are of uniform size in a mixture whose average composition is equiatomic. $T = 1000 \text{ K}$.

in energy as the distance between a pair of atoms is decreased from infinity to an equilibrium separation, which for a pair of A atoms is written $-2\epsilon_{AA}$. From standard theory for atomic solutions, the molar enthalpy of mixing is given by:

$$\Delta H_M \approx N_a z (1-x) x \omega \quad \text{where} \quad \omega = \epsilon_{AA} + \epsilon_{BB} - 2\epsilon_{AB} \quad (4)$$

where z is a coordination number.

However, for particles which are not monatomic, only those atoms at the interface between the A and B particles will feel the influence of the unlike atoms. It follows that the enthalpy of mixing is not given by equation 4, but rather by

$$\Delta H_M = z N_a \omega \ 2\delta S_V \ x(1-x) \quad (5)$$

where S_V is the amount of $A-B$ interfacial area per unit volume and 2δ is the thickness of the interface, where δ is a monolayer of atoms.

A further enthalpy contribution, which does not occur in conventional solution theory, is the structural component of the interfacial energy per unit area, σ :

$$\Delta H_I = V_m S_V \sigma \quad (6)$$

where V_m is the molar volume.

Both of these equations contain the term S_V , which increases rapidly as the inverse of the particle size m . The model predicts that *solution formation is impossible* because the cost due to interfaces overwhelms any gain from binding energies or entropy. And yet solutions do form, so there must be a mechanism to reduce interfacial energy as the particles are divided. The mechanism is the reverse of that associated with precipitation (Figure 8). A small precipitate can be coherent but the coherency strains become intolerable as it grows. Similarly, during mechanical alloying it is conceivable that the particles must gain in coherence as their size diminishes. The milling process involves fracture and welding of the attrited particles so only those welds that lead to coherence might succeed.

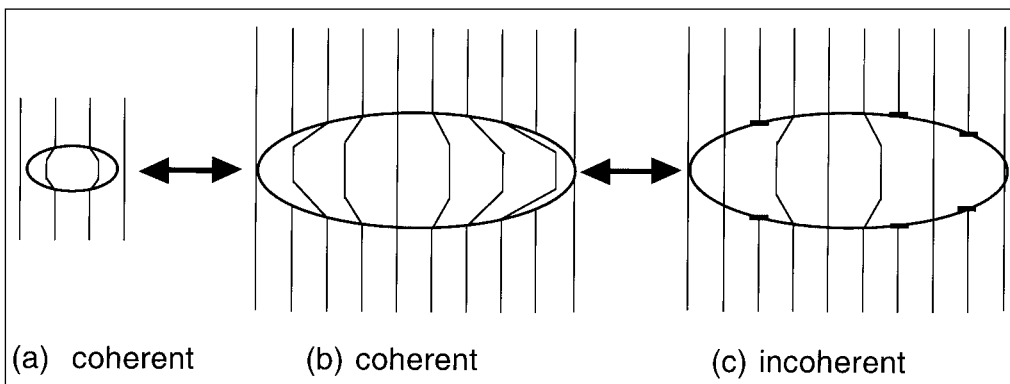


Fig. 8. The change in coherence as a function of particle size. The lines represent lattice planes which are continuous at the matrix/precipitate interface during coherence, but sometimes terminate in dislocations for the incoherent state. Precipitation occurs in the sequence $a \rightarrow c$ whereas mechanical alloying is predicted to lead to a gain in coherence in the sequence $c \rightarrow a$.

Alloy	Stored Energy J g ⁻¹
MA957	1.0
MA956	0.4
MA956 sheet	≈0.4†
MA6000	0.6
MA760	1.0
MA758	0.3

Table 3. Enthalpy of recrystallisation (Murakami *et al.*, 1993; Chou & Bhadeshia, 1993, 1994; Sha & Bhadeshia, 1994).

† In MA956 sheet, the stored energy is released over a relatively large range of temperatures and is difficult to measure accurately. For MA758 the stored energy is small and recrystallisation occurs close to the melting point, making it difficult to measure.

Another unexpected result is obtained on incorporating a function that allows the interfacial energy to decrease as the particle size becomes finer during mechanical alloying. Thermodynamic barriers are discovered to the formation of a solution by the mechanical alloying process (Badmos & Bhadeshia, 1997). When the enthalpy of mixing is either zero or negative, there is a single barrier whose height depends on the competition between the reduction in free energy due to mixing and the increase in interfacial energy as the particles become finer until coherence sets in. When the atoms tend to cluster, there is a possibility of two barriers, the one at smaller size arising from the fact that atoms are being forced to mix during mechanical alloying.

Recrystallisation temperature

One of the most intriguing features of the alloys discussed here is the fact that recrystallisation occurs at exceptionally high homologous temperatures, of the order of 0.9 of the melting temperature (T_M). This contrasts with ordinary cold-deformed metals which recrystallise readily at about 0.6 T_M , even though the mechanically alloyed variants contain more stored energy (Table 3).

Early work on mechanically alloyed ODS nickel-base superalloys (Nakagawa *et al.*, 1998) attributed the high recrystallisation temperatures to the presence of γ' precipitates. However, there are alloys for which the $-\gamma'$ dissolution temperature is below that at which recrystallisation occurs (Mino *et al.*, 1987, 1992; Kusunoki *et al.*, 1990). Furthermore, the

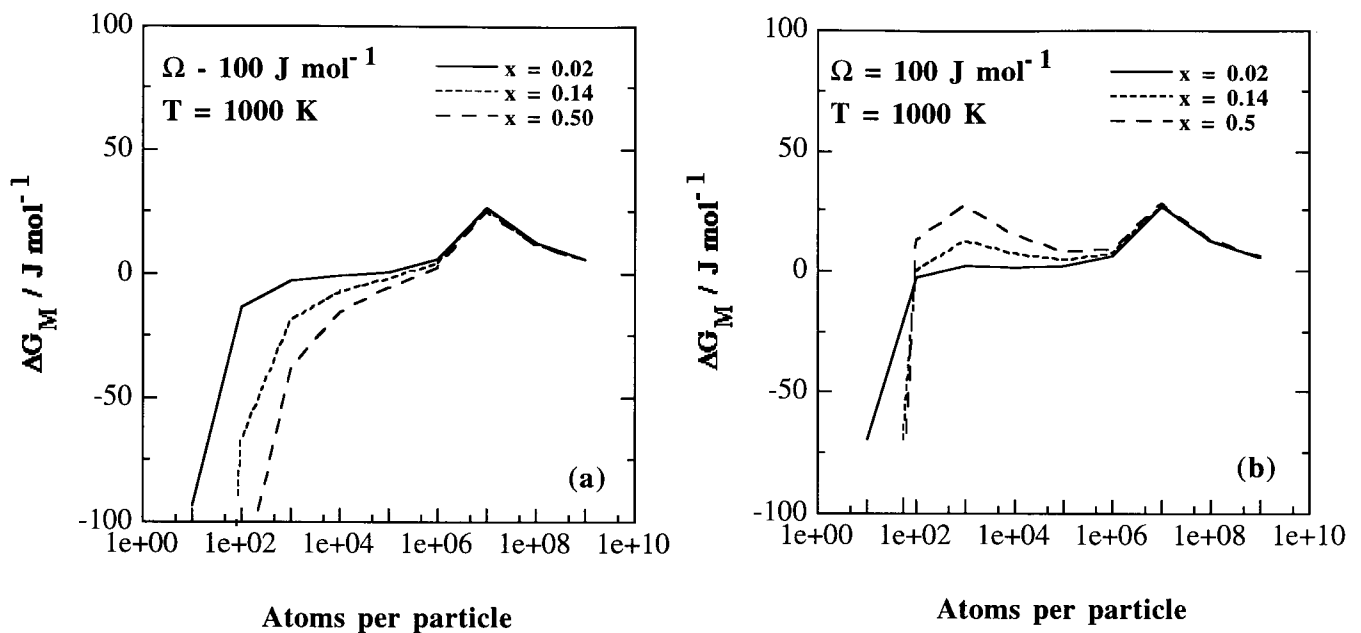


Fig. 9. Thermodynamic barriers to solution formation. (left) Case where the enthalpy of mixing is negative, i.e. unlike atoms attract. (right) Case where there is a tendency to cluster with a positive enthalpy of mixing. After Badmos & Bhadeshia (1997).

iron-base alloys do not contain any γ' and yet also recrystallise at similarly high temperatures.

It has been speculated (Jongenburger, 1988) that recrystallisation occurs when the grain boundary mobility rises suddenly when solute drag is overcome at high temperatures. This is inconsistent with the fact that the recrystallisation temperature can be reduced by many hundreds of Kelvin by a slight additional inhomogeneous deformation (Regle & Alamo, 1993; Chou & Bhadeshia, 1997).

The fine particles of yttrium oxide may interfere with recrystallisation but this does not explain why the limiting grain size following recrystallisation is enormous. In any case, recrystallisation is found to be insensitive to the overall pinning force (Murukami, 1993).

Almost all of these difficulties are resolved when nucleation is considered in detail (Murukami, 1993; Bhadeshia, 1997; Sha & Bhadeshia, 1997). It turns out that the activation energy for *nucleation* is very large. This is because the alloys have an unusually small grain size prior to recrystallisation. Recrystallisation nucleates by the bowing of grain boundaries, a process which for conventional alloys is straightforward since the distance between grain boundary junctions is usually larger than that between other strong pinning points. With the sub-micrometre grain size of mechanically alloyed metals, the grain junctions themselves act as severe pinning lines for grain boundary bowing (Figure 10). It is easy to demonstrate that this should lead to an enormous activation energy for the nucleation of recrystallisation, many orders of magnitude larger than the activation energy associated with self-diffusion (Murukami, 1993; Bhadeshia, 1997; Sha & Bhadeshia, 1997). The activation energy can be reduced dramatically if just a few grains happen to be slightly larger than others (either because adjacent grains are similarly orientated or because of local variations due to the uncertainties in the mechanical alloying process).

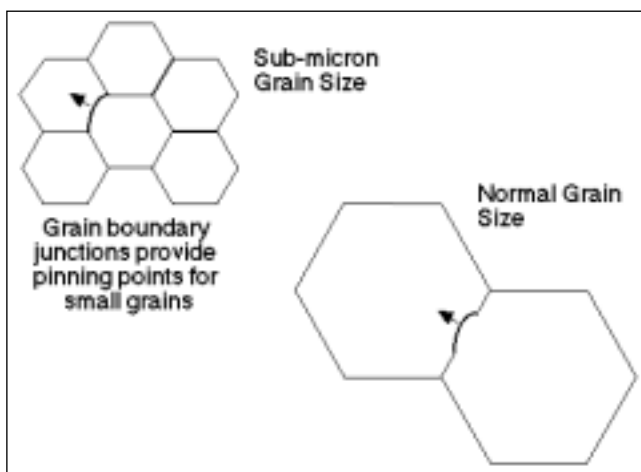


Fig. 10. The nucleation of recrystallisation occurs by the formation of a grain boundary bulge. This can occur with less constraint when the grain junctions are spaced at distances greater than the critical bulge size. With the ultra-fine grains of mechanically alloyed metals, the grain junctions are themselves pinning points, making it very difficult to form large enough bulges.

Summary

Commercial mechanically alloyed metals are fascinating in that they have helped reveal many new phenomena, some of which have yet to be investigated experimentally. Amongst the latter is the prediction that there are one or more barriers to the formation of a solid solution by a process in which the component particles are successively refined in size. A second prediction, which could be verified using detailed microscopy, is that there must be a gain in coherence as the mixture of powders approaches an atomic solution. One problem which appears to have been solved is the strange

recrystallisation behaviour; the ultrafine and uniform grains of the starting microstructure do not behave independently and hence prevent recrystallisation until temperatures close to melting.

Acknowledgments

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