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Atomically disordered nanocrystalline Co_2Si by high-energy ball milling

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Abstract. The initially ordered intermetallic compound Co_2Si with orthorhombic structure was mechanically milled in a high-energy ball mill. The structural changes with milling time were followed by x-ray diffraction, high-field magnetization measurements and differential scanning calorimetry (DSC). Ball milling results in a continuous increase of the magnetization at 4.2 K in a field of 21 T with milling time. This gives evidence that by mechanical milling anti-site disorder is generated in Co_2Si , similar to the case of orthorhombic Co_2Ge . X-ray diffraction reveals that the number of diffraction peaks decreases with increasing milling time and that all diffraction peaks broaden. The absence of a large number of diffraction lines supports the creation of atomic disorder. The average crystallite size derived from the line width shows a drastic decrease in the early stage of milling and tends to become constant with a value of 8–10 nm upon further milling. The material remains in the same structure as the starting compound even after prolonged periods of milling. Exothermic heat effects due to both atomic reordering and nanocrystallite growth are evident in the DSC scans of ball-milled samples. Well defined atomically disordered nanocrystalline Co_2Si is produced by ball milling. Since the amount of heat released in reordering and grain growth is comparable, it can be concluded that both anti-site disorder and grain boundaries are the important sources of energy storage in Co_2Si during high-energy ball milling.

1. Introduction

Mechanical milling in a high-energy ball mill has attracted much interest in recent years since it can be used as a non-equilibrium processing tool. It has been realized that ball milling of crystalline intermetallic compounds (mechanical milling, MM), or of crystalline elemental powder mixtures (mechanical alloying, MA), can be used to synthesize various metastable structures. Typical examples of these structures are amorphous materials [1–5], extended solid solutions [6], solid solutions of immiscible systems [7, 8], nanocrystalline materials [9–11] and quasicrystalline materials [12, 13]. In some cases, for instance in Ni_3Sn_2 and Co_3Sn_2 , mechanically induced transformations from a complex low-temperature crystal structure (LTP) to a simpler high-temperature crystal structure (HTP) have also been found [14–16]. The non-equilibrium phase transformation LTP→HTP was accompanied by the creation of a special type of atomic disorder, namely ‘redistribution of transition metal atoms over two different types of transition-metal site’. It was found that this particular type of disorder cannot be induced by rapid quenching from high temperatures. Such a disorder was also generated by ball milling in ferrimagnetic Mn_3Sn_2 [17] and ferromagnetic Fe_3Ge_2 [18]. Both Mn_3Sn_2 and Fe_3Ge_2 have a pure B8_2 hexagonal structure, the same structure as the HTP of Ni_3Sn_2 and Co_3Sn_2 . This structure extends in the phase diagrams up to the melting temperature. In the B8_2 structure there are two different types of interstice that can

be occupied by the transition-metal atoms: octahedral (I) and tetrahedral (II) interstices. The LTP of Ni_3Sn_2 and Co_3Sn_2 is similar to the HTP, only the atoms are shifted somewhat to form an orthorhombic structure. In the equilibrium state, in all T_3X_2 compounds (T = transition metal, X = non-transition metal) the I sites are fully occupied, whereas the II sites are only half-occupied. Ball milling of the T_3X_2 compounds resulted in a transfer of the T atoms from I to II sites i.e. to the above-mentioned particular type of disorder. This disorder did not induce amorphization in all the studied T_3X_2 compounds even after milling for prolonged periods. In a previous paper [19], we reported a detailed study of the compound Co_2Ge upon ball-milling in the same mill as was used in [14–18]. Co_2Ge is very similar to Ni_3Sn_2 and Co_3Sn_2 in that the compound also crystallizes in the orthorhombic structure at room temperature (LTP, $\alpha\text{-Co}_2\text{Ge}$), while above 625°C it transforms to a high-temperature phase (HTP, $\beta\text{-Co}_2\text{Ge}$) with the B8₂ hexagonal structure, which melts at much higher temperatures. However, in occupation of sites Co_2Ge is different from T_3X_2 compounds. This is due to the composition. In Co_2Ge both I and II sites are fully occupied, whereas in T_3X_2 compounds the II sites are only half occupied as mentioned above. Because of the full occupancy of the interstices a different type of disorder is to be expected in Co_2Ge than in the T_3X_2 compounds. Indeed, ball milling of both $\alpha\text{-Co}_2\text{Ge}$ (LTP) and $\beta\text{-Co}_2\text{Ge}$ (HTP) resulted in anti-site disorder, i.e. substitution of both atomic species on each other's sublattice. In T_3X_2 compounds the structural frame of the X atoms is preserved during ball milling. In contrast, by anti-site disorder the structural frame is destroyed in Co_2Ge , which makes the LTP \rightarrow HTP transformation impossible and results eventually in amorphization. Moreover, the amorphous Co_2Ge thus obtained shows spin-glass behaviour [19,20]. Amorphous Co_2Ge could not be generated by traditional melt spinning. It was the first spin-glass phase synthesized by mechanical milling reported. In order to obtain more insight about the mechanisms of the structural changes in B8-like compounds by ball milling, we studied Co_2Si upon milling. Co_2Si was chosen because its orthorhombic structure is the same as that of the $\alpha\text{-Co}_2\text{Ge}$ (LTP). This implies that ball milling of Co_2Si may also result in anti-site disorder and finally in amorphization. The milling process was examined by x-ray diffraction, magnetic measurements and differential scanning calorimetry (DSC).

2. Experimental details

The intermetallic compound Co_2Si was obtained by arc melting of weighed amounts of pure Co and Si in a purified Ar atmosphere. Arc melting was repeated at least four times in order to obtain a homogeneous sample. The arc-melted button was crushed to powder and annealed at 850°C for 2 d and at 500°C for 3 d. The x-ray diffraction pattern of the annealed sample shows single phase material with the orthorhombic structure. Mechanical milling was carried out in a hardened steel vacuum vial (inner diameter 6.5 cm) with a tungsten carbide bottom. Inside the vial, a hardened steel ball with a diameter of 6 cm was kept in motion by a vibrating frame (Fritsch: Pulverisette 0), upon which the vial was mounted. In order to prevent reactions with O_2 or N_2 , the milling was performed under continuous pumping. During the milling the vacuum was kept at a level of about 10^{-6} Torr. The starting amount of material was a few grams. X-ray diffraction powders were taken from the samples milled for different periods and after x-ray diffraction the powder was used for high-field magnetization measurement and for DSC analysis.

The x-ray diffraction patterns were taken by means of a Philips diffractometer with vertical goniometer using $\text{Cu K}\alpha$ radiation. The high-field magnetization measurements were performed at 4.2 K in the Amsterdam High Field Installation [21] in which magnetic

fields up to 40 T can be generated in a semi-continuous way. A stepwise field profile up to 21 T was used. The sensitivity of this magnetometer is about 10^{-5} A m². Differential scanning calorimetry (DSC) was carried out in a Perkin-Elmer DSC-7 in an argon flux at a speed of 30 cc min⁻¹ to protect the sample against oxidation. A heating rate of 10 K min⁻¹ was used. The scan was repeated twice for each sample. After the first scan, a second scan was made immediately without changing the sample so that the measurement was done under the same conditions. The second scan was used as the baseline for calculating the enthalpy released. The temperature and the reaction heat were calibrated by pure In and Zn standards.

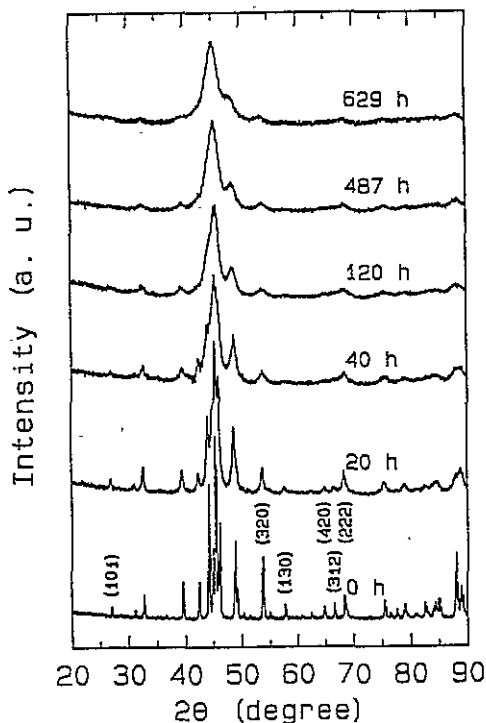


Figure 1. Typical x-ray diffraction patterns of Co_2Si after various periods of milling.

3. Results and interpretation

3.1. Structure and thermal stability

The typical x-ray diffraction patterns of Co_2Si for various milling times are given in figure 1. In agreement with the literature [22], all the diffraction peaks of the starting compound (the curve 0 h) can be identified as a single phase having the ordered orthorhombic structure with space group $Pnma$. Some of the diffraction peaks, the behaviour of which will be discussed below, are indexed explicitly in figure 1. During ball milling the intensity of all diffraction peaks decreases gradually with milling time. Meanwhile, the width of the Bragg peaks increases upon milling. After 20 h of milling, some of the low-intensity diffraction peaks disappear and the Bragg peaks around $2\theta = 45^\circ$ become overlapping. After 120 h of milling many diffraction peaks including the relatively strong diffraction lines, e.g. the (420) and (312) lines, disappear and it is no longer possible to separate the diffraction

peaks around $2\theta = 45^\circ$. Upon further milling a continued decrease of the diffraction line intensities is observed. After 487 h of milling, the diffraction lines (101) and (130) disappear and broaden further. After 629 h of milling, changes in x-ray diffraction pattern are still observable, e.g. a further decrease of all diffraction line intensities takes place, but the pattern is still characteristic of the orthorhombic structure. Amorphization does not occur after this period of milling. The absence of a large number of diffraction peaks strongly indicates that atomic disorder is generated in Co_2Si by mechanical milling.

The application of x-ray diffraction line broadening to study the structure of plastically deformed metals has been extensively reviewed by several authors and is a standard experimental method (see for instance [23] and [24]). The major sources for line broadening from cold-worked materials are believed to be (i) small crystallite size (coherent domains) and (ii) strains and distortions within the crystallites. The size of crystallites can be estimated from the well known Scherrer equation $d = k\lambda/(\beta \cos \theta)$, which can give reasonable values for the dimensions of crystallites in the range from 1 to 100 nm. Here k is a constant with a value close to unity but dependent on crystallite shape and hkl reflection, λ is the wavelength, θ the diffraction angle and β the integral breadth of an observed profile. Using Langford's Voigt-deconvolution method (see [25] for details) the internal strain broadening was subtracted. The x-ray diffraction pattern of the starting compound was used as a standard to correct for the instrumental broadening. During evaluation, the k value was chosen as unity and the (320) and (222) reflections were used. The average crystallite size of Co_2Si milled for various periods thus obtained is plotted in figure 2 as a function of milling time. It can be seen that the average crystallite size decreases abruptly with increasing milling time up to 80 h of milling. Upon further milling it decreases slightly and tends to saturate with a value of 8–10 nm. So, during mechanical milling Co_2Si becomes nanocrystalline.

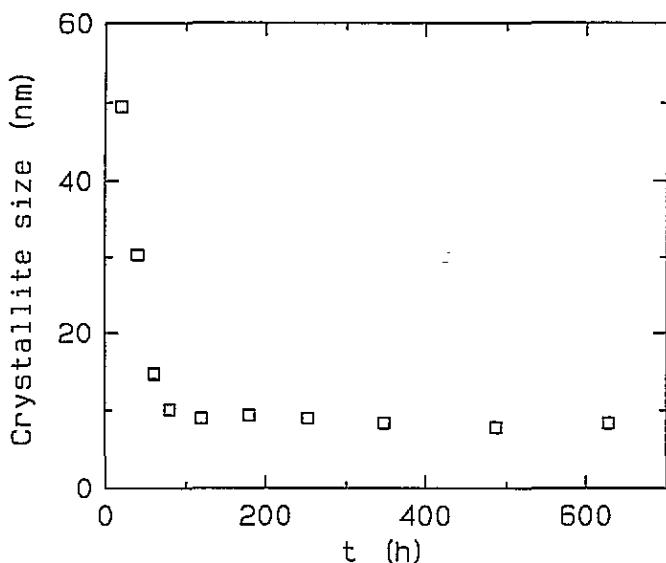


Figure 2. The average crystallite size of Co_2Si as a function of milling time.

The thermal stability of Co_2Si after various periods of milling was analysed by DSC. For almost all the ball-milled samples three exothermic heat effects are evident. The transition shape, the transition heat and the peak temperature are very sensitive to the milling time. Three typical DSC scans of Co_2Si milled for 40 h, 120 h and 629 h are given in figure 3.

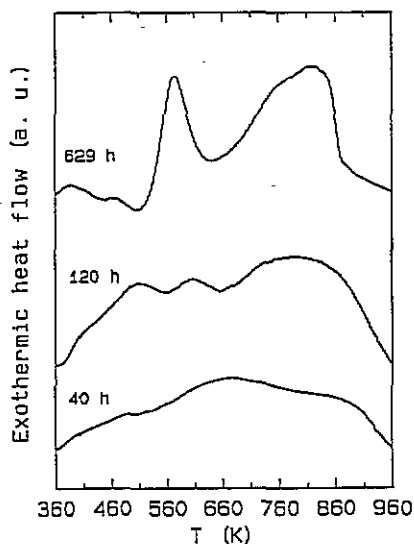


Figure 3. Typical DSC scans of Co_2Si after milling for 40 h, 120 h and 629 h.

From this figure a broad exothermic heat effect between 360 K and 960 K is observed in the sample milled for 40 h. This broad transition in fact consists of three overlapping exothermic peaks, which becomes more manifest in the DSC scan of the sample milled for 120 h. After long milling time, e.g. 629 h, the second and the third transitions become much more pronounced and better separated. The peak temperature of both the second and the third peaks shifts to lower temperature with increasing milling time. The total heat evolved in these transitions is derived and plotted in figure 4 as a function of milling time. The released heat first rapidly increases with milling time up to a period of 80 h and then increases more slowly upon further milling. It does not saturate even after prolonged period of milling, e.g. milling for 629 h. The observed tendency is similar to the change of the magnetization (see figure 7).

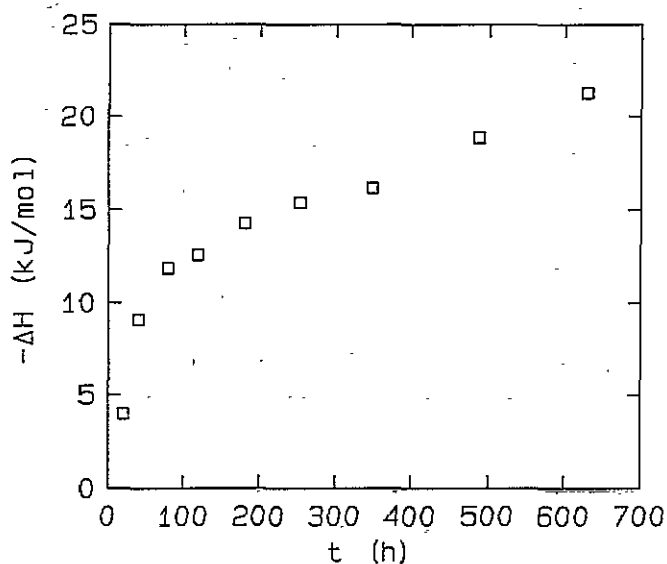


Figure 4. The total heat release during atomic reordering and crystallite growth as a function of milling time.

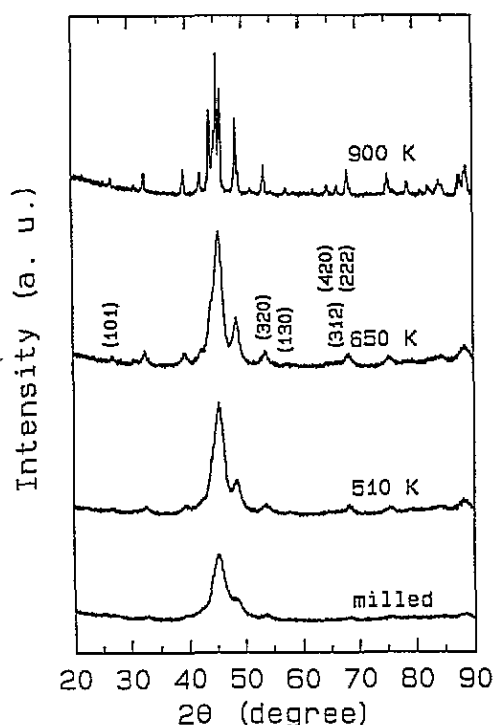


Figure 5. X-ray diffraction patterns of Co_2Si milled for 629 h after heating to 510 K, 650 K or 900 K in the DSC and subsequently rapidly quenching to room temperature.

For a better understanding of these exothermic heat effects, the sample milled for 629 h was heated in the DSC to the various temperatures where the heat effects occur, and subsequently rapidly cooled to room temperature. Figure 5 gives the x-ray diffraction patterns of the sample after heating to 510 K (end of the first exothermic effect), 650 K (end of the second exothermic peak) and 900 K (end of the third exothermic peak). The pattern for the ball-milled sample is also included for comparison. The pattern labelled 510 K shows somewhat sharper diffraction peaks and a weak separation of the diffraction peaks around $2\theta = 45^\circ$. This indicates that the first exothermic effect is attributable to irreversible defect annihilation and strain release. The pattern labelled 650 K shows a number of extra diffraction peaks such as (101), (130), (420) and (312) lines. The crystallite size derived from (320) and (222) lines shows a slight increase from 8 to 14 nm. This means that the second peak mainly results from the reordering process of the atomically disordered Co_2Si . The pattern labelled 900 K shows that all the diffraction peaks are very sharp and that the relatively low-intensity diffraction peaks are visible. The average crystallite size estimated from this pattern is about 50 nm. So, the third pronounced exothermic peak is mainly due to the growth of nanocrystallites. This again proves that well defined nanocrystalline Co_2Si is produced by ball milling.

The observation of three exothermic peaks in the heating process of ball-milled Co_2Si can be understood as follows. Theoretically, when a fully disordered intermetallic compound is heated, three major exothermic heat effects are expected: (i) the relaxation of lattice defects and stresses; (ii) the development of atomic reordering and (iii) crystallite growth from the nanocrystalline state to the microcrystalline state. In view of the required diffusion distances, these processes are expected to occur in the following sequence: (i)→(ii)→(iii). Similar phenomena were also observed in mechanically disordered Ni_3Al by Baró *et al* [27] and in $\text{Ni}_3(\text{Fe}, \text{Al})$ by Yavari [28] as well as in disordered Mn_3Sn_2 and Fe_3Ge_2 [18, 26].

However, the three processes do not always result in three separate peaks. Furthermore, the third exothermic heat effect in the ball-milled Co_2Si is similar to that measured by Eckert *et al* [10] during nanograin growth in ball-milled nickel. However, the third stage is not always observable in the DSC scans. That it is found in all ball-milled samples of Co_2Si indicates that well defined nanometre-scale crystallites are formed by heavy mechanical deformation. The decrease of the peak temperature with increasing milling time indicates a decrease of crystallite size [29]. The reordering temperature also shifts to lower temperature indicating that disordering decreases the activation energy of the reordering process in Co_2Si . Since the transition heats evolved in the atomic reordering and in the grain growth are comparable in value, we can conclude that both atomic disorder and nanocrystallite boundaries are major sources of mechanical energy storage.

3.2. High-field magnetization measurements

Typical high-field magnetization curves at 4.2 K up to 21 T are presented in figure 6. It is clear that both the magnetization and the magnetic behaviour of the ball-milled Co_2Si are quite different from those of the starting compound. The magnetization at 4.2 K of the ball-milled samples increases significantly with milling time. The magnetization of the various samples at 4.2 K taken at 21 T is plotted in figure 7 as a function of milling time. It shows the same tendency as the released heat (figure 4), i.e. the magnetization first abruptly increases with milling time up to a period of 80 h and then gradually increases upon further milling. It does not saturate even after 629 h of milling.

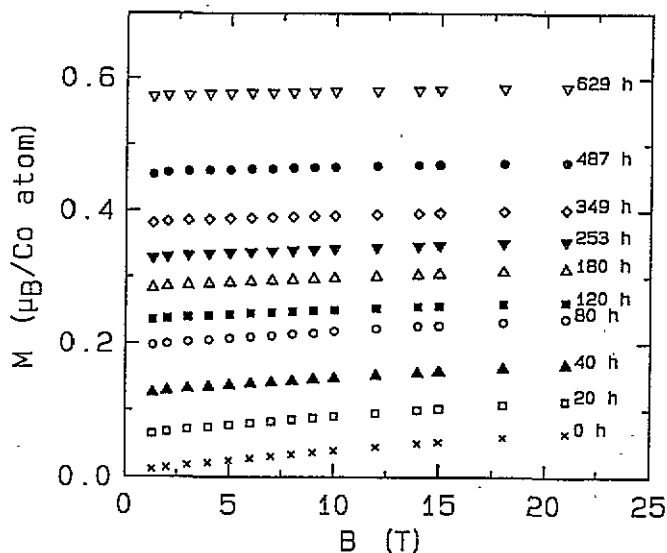


Figure 6. Typical high-field magnetization curves of Co_2Si at 4.2 K after various periods of milling.

The continuous increase of magnetization with milling time can well be understood in terms of atomic disorder. In previous investigations [30, 31, 17–19] it was demonstrated that atomic disorder is generated by mechanical milling. The type of atomic disorder turned out to be characteristic of the specific compound. It has been discussed in detail in [19] that if the atomic sizes of the different species of atoms in a fully occupied (no vacant lattice sites) crystalline structure are quite close mechanical milling may induce anti-site atomic disorder. As mentioned in section 1, in the crystal structure of Co_2Si Co atoms occupy two different types of lattice site (octahedral (I) and tetrahedral (II) interstices), and both I and

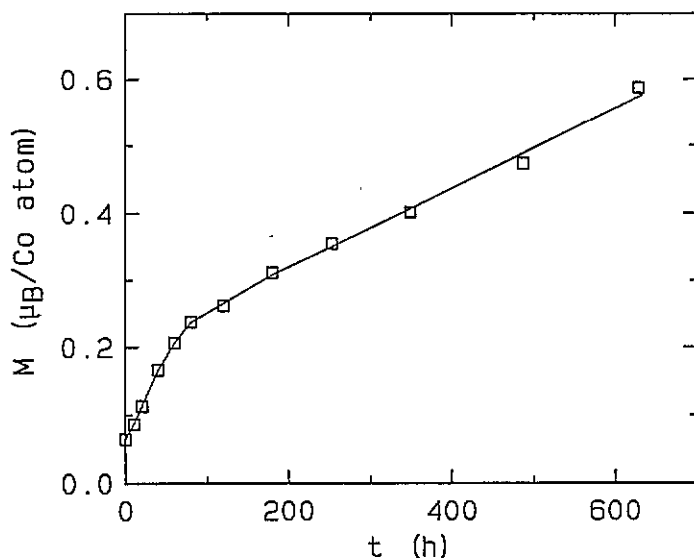


Figure 7. Magnetization of Co_2Si at 4.2 K and 21 T as a function of milling time.

II sites are fully occupied. Although the size difference between Co and Si atoms is slightly larger than that between Co and Ge atoms, under mechanical impact anti-site disorder can still be introduced in Co_2Si since no vacant lattice sites are available in the structure. This means that during mechanical milling Co and Si atoms will exchange positions. Such an exchange will lead to a variation of the neighbourhood of a Co atom. It is clear from figure 7 that the average magnetic moment of Co atoms in the ordered crystalline state of Co_2Si is approximately $0.06\mu_B/\text{Co atom}$, which is much less than $1.7\mu_B/\text{Co atom}$, the value for pure Co [32]. The increase in magnetization can then be interpreted as follows. In Co_2Si Si is a non-magnetic element. This means that only the coordination of the Co atoms determines the formation of the magnetic moment. In the starting compounds, the Si atoms surrounding a Co atom hinder the Co–Co interaction, which limits the formation of a large magnetic moment. During mechanical milling, due to the exchange of Co and Si atoms, the neighbourhood of a Co atom will change. In this way clustering is favoured with more and more Co atoms surrounding a Co atom. Such a Co-rich cluster will bear a larger magnetic moment because of an increasing probability of Co–Co exchange interactions. The number of the Co-rich clusters will gradually increase with increasing milling time. This will lead to an increase of the average magnetization. Therefore, it is observed that the magnetization of Co_2Si continuously increases with increasing milling time. Thus, the continuous increase of the magnetization gives evidence that by mechanical milling anti-site disorder is generated in Co_2Si as it is in Co_2Ge [19].

To check the influence of impurities such as Fe, which may be introduced during ball milling, we also measured the magnetization curves of some typical ball-milled samples after annealing in the DSC. It turned out that both the magnetization and the magnetic susceptibility of the annealed samples are quite close to those of the unmilled sample. Thus, the increase of magnetization really results from anti-site disorder in Co_2Si , while the influence of impurity Fe is negligible.

4. Discussion

From the previous section it appears that ball milling creates anti-site disorder in Co_2Si and decreases the crystallite size. Disorder in Co_2Si is evidenced by all techniques used in the

present investigation. The usual method to monitor disordering in intermetallic compounds is measurement of the change of the long-range-order parameter by comparing the relative intensities of superlattice reflections and fundamental lines. However, it is not always easy to distinguish the superlattice reflections from the diffraction peaks in compounds with a complex structure such as, in the present case, the orthorhombic structure. It has been shown that the change of the superconducting transition temperature in superconducting compounds or of the magnetization in magnetic materials can be a good measure of the degree of order upon ball milling or quenching. In the present study we again emphasize that the use of magnetic measurements is very effective to monitor the structural change during non-equilibrium processes such as high-energy ball milling. From the change of x-ray diffraction patterns we know that a large number of diffraction lines disappears after ball milling. This gives an indication of the disordering by milling. During heating of ball-milled samples a pronounced exothermic heat effect corresponding to the reordering process is observed. This confirms the occurrence of disordering. The information obtained from x-ray diffraction and DSC does not give the type of disorder. However, the continuous variation of magnetization leads at least to the tentative conclusion that anti-site disorder is generated in Co_2Si by ball milling. The disordering process is faster in the beginning and slower in the intermediate stage.

The crystallite size decreases rapidly in the early stage of milling and decreases slightly upon further milling. After long milling time, the crystal dimensions are of the order of 8–10 nm and further refinement seems difficult to achieve. This is due to the fact that the small crystallite size itself provides a limit for the glide of dislocations and therefore for further crystallite size refinement, since the forces required to maintain plastic deformation, via dislocation movement, may be very large in a polycrystalline material with decreasing crystallite size [9]. In contrast, plastic deformation of nanocrystalline material seems to occur at considerably lower stress via gliding along the grain boundaries, as recently has been shown for nanocrystalline ceramics [33].

From the above results and discussions, it is clear that mechanical milling of Co_2Si in a high-energy ball mill generates anti-site disorder. This is similar to the case of Co_2Ge . Moreover, well defined nanocrystalline states are also produced in Co_2Si . However, the material does not transform to the amorphous form even after 629 h of milling. This is different from what occurred in Co_2Ge . It is likely to be due to the fact that the milling time is not long enough. Co_2Si remains in the original crystal structure but in a metastable state. This is clear from figures 4 and 7, where neither released heat nor magnetization saturated even after 629 h of milling. This means that during further milling mechanical energy can still be stored by the continued creation of anti-site disorder. Since crystallite size is not being refined further, the grain boundaries cannot store more energy.

5. Conclusions

Mechanical milling of the initially ordered intermetallic compound Co_2Si results in anti-site disorder and the formation of nanocrystalline material. X-ray diffraction patterns show an absence of a large number of diffraction lines and a broadening of the remaining diffraction peaks after ball milling. The continuous increase of magnetization (eventually by a factor as high as ten) confirms the generation of anti-site disorder in Co_2Si . The observation of pronounced exothermic heat effects for both atomic reordering and crystallite growth strongly supports the conclusion that atomic disorder is generated in Co_2Si by ball milling and that nanocrystalline material is produced. The average crystallite size is about 8–10 nm

after long milling time. The total heat released during the reordering and crystallite growth is about 22 kJ mol⁻¹. Both anti-site disorder and grain boundaries are major sources of energy storage during ball milling. The use of magnetic measurements for monitoring the structural changes in intermetallic compounds during non-equilibrium processing such as ball milling is again emphasized.

Acknowledgment

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