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Evolution of the microstructure in carbon nanotube reinforced Nickel matrix composites processed by high-pressure torsion

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Abstract. Carbon nanotube (CNT)-reinforced nickel matrix composites were processed using high-pressure torsion (HPT) at room temperature (RT). Different CNT weight fractions were used in order to study the behavior of the composites in the "as sintered" and the "as deformed" conditions and to determine the effect of the amounts of CNT added on the different processing methods. The samples were analyzed by means of Vickers microhardness and electron microscopy. According to the results, increasing the CNT content in the "as sintered" condition increases the agglomerate size but decreases only slightly the grain size. Regarding the "as deformed" condition it showed little to negligible effect in further refining the microstructure. By means of HPT the hardness was increased up to 800%. It was concluded that the microstructure could be further improved in terms of grain size and agglomerate size and distribution by means of HPT.

1. Introduction

Seeking the stabilization of the microstructure against grain growth is critical in nanocrystalline (NC) and ultrafine-grained (UFG) materials processed by severe plastic deformation (SPD), since they possess high amounts of stored energy in their large grain boundary area, giving them an unstable nature. This instability can activate microstructural recovery and grain growth processes even at low temperatures that can alter the material properties considerably. In some studies, the addition of reinforcing phases to metallic matrices subjected to SPD has been performed with the aim of overcoming this issue and given a proper distribution of the particles [1] they even result in enhanced mechanical properties [2, 3]. The resulting metal matrix composites (MMC) behave differently than pure metals and provided that the appropriate reinforcing phase is chosen and a uniform distribution of the agglomerates can be obtained, the thermal instability drawback might be overcome by making use of different mechanisms (e.g. by hindering the grain boundary and dislocation mobility).

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In this study, we propose the use of carbon nanotubes as reinforcing phase in Ni matrix composites, acting a stabilizing second phase. The choice is based on the fact that, though they possess outstanding intrinsic properties, they have also shown the ability to pin the grain boundaries under thermal inputs [4, 5]. The results presented here are emphasized on the comparison between the "as sintered" and the "as deformed" conditions of the studied composites and serve as a starting point of a deeper microstructural study, which will be discussed elsewhere.

2. Experimental

Carbon nanotube (CNT)-reinforced nickel matrix composites were processed using high-pressure torsion (HPT). Different CNT weight fractions were studied, namely: 0.5 wt.% (2.4 vol.%), 1 wt.% (4.8 vol.%), 2 wt.% (9.5 vol.%) and 3 wt.% (14.5 vol.%). These composites were sintered by means of hot uniaxial pressing (HUP) and afterwards subjected to HPT at room temperature (RT) using different number of turns (1T, 4T, 10T and 20T) under 4 GPa of pressure.

The "as sintered" condition was analyzed by means of Vickers micro-hardness, scanning electron microscopy (SEM), with a resolution of 125 nm per pixel, and electron backscattered diffraction (EBSD). The electron microscopy characterization was performed on a dual beam system Helios NanoLab 600 (FEI). The EBSD measurements were performed with an EDAX TSL detector, with a 20 kV voltage bias and 22 nA current. The step size for the analysis was varied for the different samples, ranging from 150 to 500 nm.

The evolution of the microstructure along the radial direction, corresponding to increasing equivalent strain values ε_{vM} [6] (according to $\varepsilon_{vM} = \frac{2\pi nr}{t\sqrt{3}}$, where n is the number of turns, t is the sample thickness and r the distance from the center of the sample), was studied by means of SEM using backscattered electron imaging (BSE) as a faster way of obtaining information about the mean grain size (equivalent diameter), given that in the "as deformed" samples, the significant residual stresses made difficult the proper sample preparation for EBSD. The shape, size and distribution of the CNT agglomerates were also studied. The images were taken every 1 mm along the radial direction with a resolution of 2.5 nm per pixel.

Vickers micro-hardness, which is extensively used as a quick way of assessing the saturation in the microstructural refinement of such composites, was performed every 0.5 mm along the radial direction, using an indenter Struers DuraScan 50/70/80.

3. Results and discussion

The mean grain size values obtained for the different compositions of the HUP samples by means of EBSD lay all within the micrometer range and are summarized in Table 1. From these values, no clear correlation between the mean grain size and the CNT concentration can be extracted. This might be due to an incomplete pinning effect of the CNTs on the grain boundaries during the densification stage [7].

Table 1. Mean grain sizes of HUP samples obtained by EBSD. For the determination of the mean and standard deviation, a log-normal distribution was assumed.

HUP Samples	Mean grain size [μm]
0.5wt.% MWCNT/Ni	4.80 ± 2.89
1wt.% MWCNT/Ni	3.10 ± 1.17
2wt.% MWCNT/Ni	3.42 ± 1.55
3wt.% MWCNT/Ni	3.67 ± 1.67

Figure 1 shows the evolution of the grain size (equivalent diameter) along the radius of the samples, obtained from BSE images for the deformed samples. Here, it was assumed that the grains are the areas delimited by the different gray values within the image, and the misorientation angle between the grains, which cannot be measured with this method, was not considered. The results show a large decrease in grain size after HPT and at early stages of the deformation, but only a subtle decrease with increasing equivalent strains reaching values down to 100 nm, which is independent of the CNT volume fraction. From this, it can be stated that the increment of the CNTs volume fraction has little to negligible effect on further refining the microstructure during deformation, presenting similar results regarding the grain size for the same processing conditions (as shown in Figure 1).

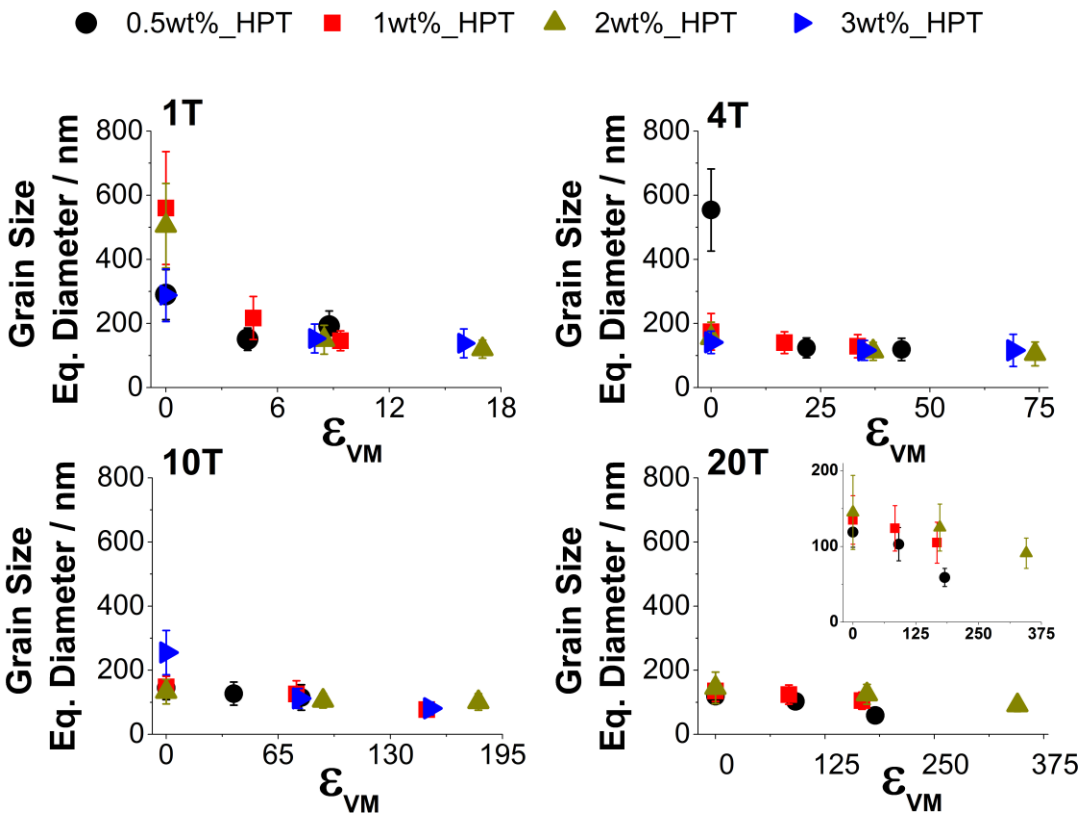

Figure 1. Change in grain size (equivalent diameter) with increasing equivalent strains. (Sample 3wt.% could not be further deformed by HPT at RT due to its high hardness causing slippage between the sample and the anvils).

Regarding the hardness, table 2 summarizes the values for the as sintered condition (HUP). The hardness increases only slightly with increasing CNT content. This correlates well to what has been previously reported in the literature [4], and is traced back to the role played by the partial CNT concentration in the dislocation mobility hindering in these composites during the indentation stage. The CNTs act as pinning points to the dislocation fronts, progressively reducing their energy. This effect combined with the microstructural refinement, leads to a combination of two strengthening mechanisms, namely: grain boundary strengthening (Hall-Petch effect) and dispersion strengthening (Orowan mechanism). Since there are no relevant changes in the mean grain size in the HUP state, the largest role in the enhancement might thus be provided by an Orowan-type mechanism.

Table 2. Vickers hardness HUP samples as a function of the partial CNT fraction.

HUP Samples	Hardness [GPa]
0.5wt.% MWCNT/Ni	1.17 ± 0.05
1wt.% MWCNT/Ni	1.17 ± 0.03
2wt.% MWCNT/Ni	1.20 ± 0.07
3wt.% MWCNT/Ni	1.31 ± 0.07

In Figure 2 the evolution of the hardness along the radial direction is shown for the HPT samples. The results show a significant increase in hardness with increasing equivalent strains of about 800 % with respect to the as sintered condition. Interestingly, even after 20 turns the hardness values continue to increase indicating that the onset of the microstructural refinement saturation has not been reached for the equivalent deformation here studied. Remarkably, all the analyzed concentrations show the similar trends in the hardening behavior. Thus, it could be stated that this feature is mainly based on the nature of the matrix and cannot be straightforwardly correlated to the amount of second phase. Nevertheless, a slight decrease in the "slope" of the "lines" is observed for increasing CNT content, which can be a "sign" that the dislocation and grain boundary mobility are hindered by the CNT during deformation.

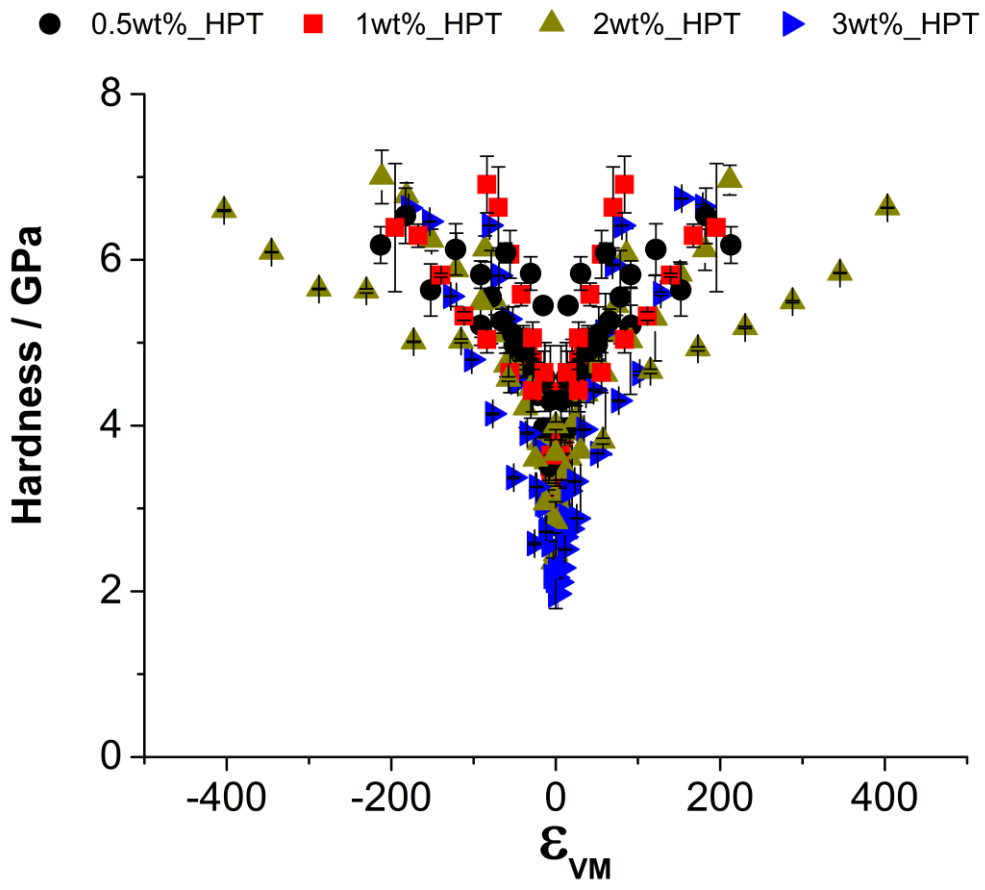

Figure 2. Variation of the hardness with increasing equivalent strains. The negative values of the equivalent strain represent only the opposite direction of the radius and do not depict a physical value.

Focusing on the effect of the deformation on the CNT agglomerate morphological characteristics, the analysis is directed to the study of the change in size and shape of the CNT clusters. Figure 3 shows binarised images for the HUP samples showing the CNT agglomerates and the corresponding mean agglomerate size values are summarized in table 3. From this observation, it becomes evident that the agglomerate size increases with increasing CNT content. This is explained by the fact that for larger CNT concentrations, there is a lower mean free path for CNT re-clustering. This has been already observed in sintered bulk metal matrix composites, and is traced back to the fact that, since the CNTs are not embedded in the metal particles, the material transport during sintering brings them closer together increasing their probability of reagglomeration due to van der Waals interactions [8].

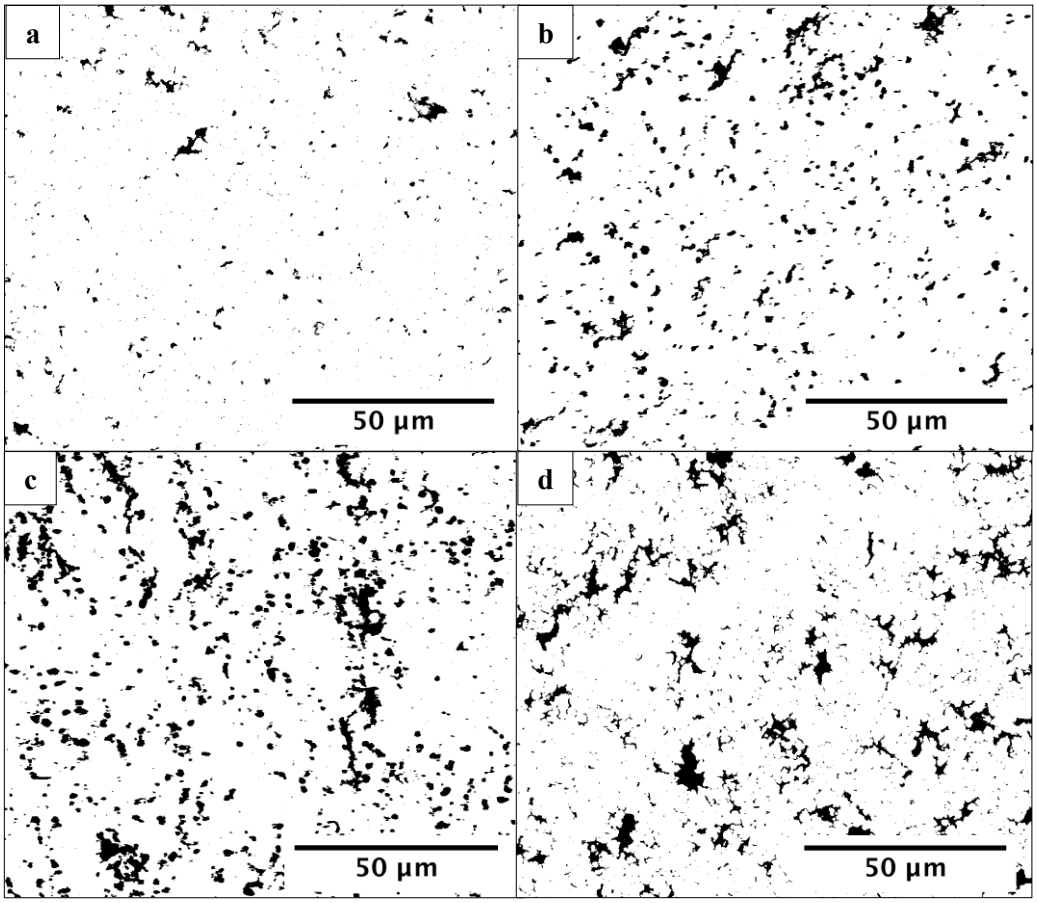

Figure 3. Binary images HUP samples. a) 0.5wt.% MWCNT/Ni, b) 1wt.% MWCNT/Ni, c) 2wt.% MWCNT/Ni and d) 3wt.% MWCNT/Ni.

Table 3. Mean agglomerate sizes of HUP samples obtained by image analysis using the image-analysis software FIJI.

HUP Samples	Mean agglomerate size [μm]
0.5wt.% MWCNT/Ni	1.91 ± 1.52
1wt.% MWCNT/Ni	2.44 ± 1.62
2wt.% MWCNT/Ni	3.11 ± 2.41
3wt.% MWCNT/Ni	3.77 ± 2.34

Concerning the as-deformed state, figure 4 presents BSE images taken at 3 mm from the center for different compositions and number of turns. The evolution of the shape, the distribution and the agglomerate size can be exemplarily visualized in this figure. The agglomerates develop an elongated shape in the shear direction at early stages of the deformation. With higher number of turns, the agglomerates become more rounded and their distribution improves. The agglomerate size decreases significantly when comparing to the as sintered condition, reaching values down to the sub-micrometer range.

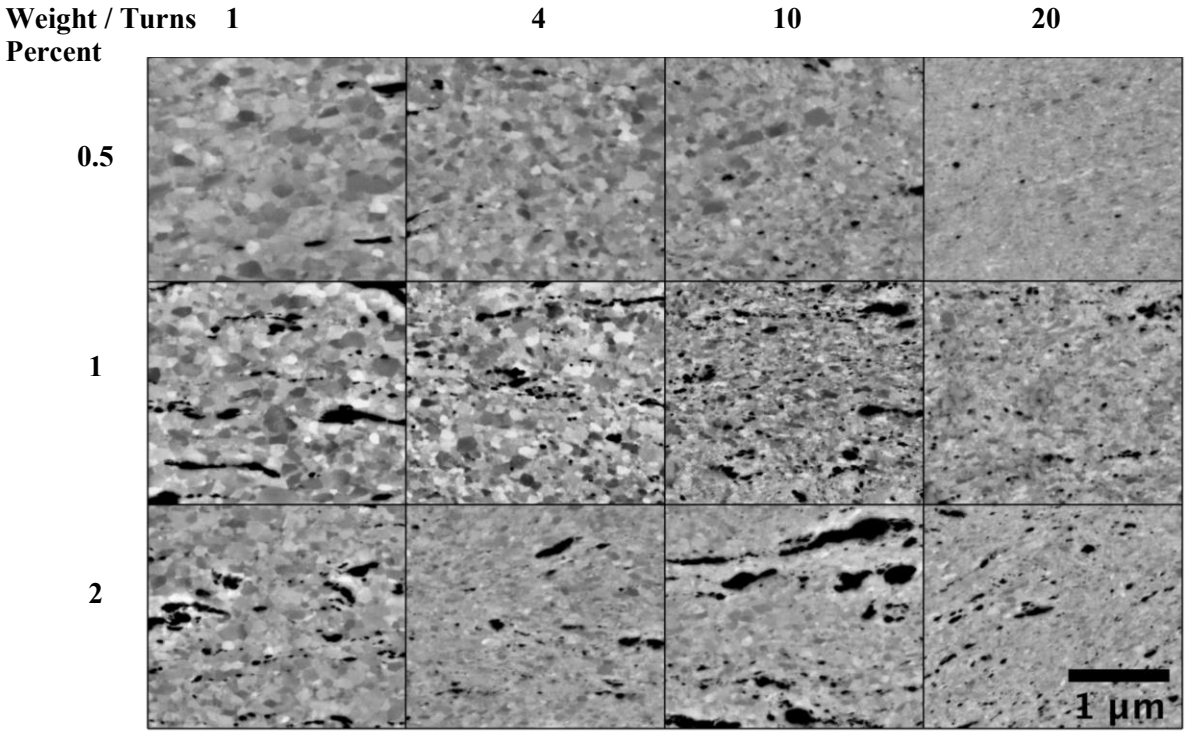

Figure 4. BSE images for a sample set at a radius of 3 mm. (Equivalent strain increases from left to right).

The decrease in grain size is within the same range and remains almost constant for the higher equivalent strains (as shown in figure 2), resembling the saturation onset of the microstructural refinement. Nevertheless, according to the Vickers hardness results, this statement cannot be confirmed for the parameters of HPT here studied. The results suggest that the microstructure can be

further refined in terms of grain size (see inset in figure 1) and improved in terms of agglomerate size and distribution by means of HPT.

4. Concluding remarks

CNT reinforced Ni matrix composites with different CNT contents were processed by HPT at RT using 4 GPa of pressure. When comparing to the HUP samples, it could be shown that after HPT not only the grain size but also the agglomerate size decreases and the microhardness increases significantly. Nevertheless, increasing the CNT content both in HUP and in HPT samples showed little to negligible effect on further refining the microstructure. Furthermore, the size and spatial distribution of the agglomerates was improved by HPT. Finally, the results suggest that the microstructure can be further improved in terms of grain size and agglomerate size and distribution by means of HPT (e.g. by optimizing the parameters used during processing).

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