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INCORPORATION OF CARBON NANOTUBES IN STAINLESS STEEL
304L VIA MECHANICAL ALLOYING

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Abstract. Carbon Nanotubes are one of the allotropic forms of carbon and, since its discovery, the scientific community seeks ways to use the unique electrical and mechanical properties of CNTs as composites, incorporated in metal matrices. Despite an increasing development in researches involving the success in incorporating carbon nanotubes in metals, such as Silicon, Copper and Aluminum, and applying in areas including welding and thermal spraying, there is barely any knowledge in the community concerning the use of stainless steel as metal matrix. This work uses the Mechanical Alloying process to incorporate CNTs in an AISI 304L stainless steel. The mixture of AISI 304L stainless steel and 5 wt.% CNTs was placed in a planetary ball mill alongside 25 stainless steel spheres of 1cm diameter each, and rotated at 200 rpm. Samples with 0 min, 30 min, 60 min and 90 min of Mechanical Alloying process were collected to be characterized using XRD, Raman and SEM. The results indicated that the 60 min sample achieved homogeneous dispersion and aggregation in the metal matrix, which qualifies such parameters as more suitable to produce the CNT-AISI 304L steel.

Keywords: Carbon Nanotubes, Stainless Steel, Mechanical Alloying, XRD, Raman.

1. INTRODUCTION

Nanotechnology has been studied over the last two decades because it supports answers to solve problems of conventional engineering limitations such as reinforcement of metallic matrix (Pradeep, 2008). In this context, carbon nanotubes (CNT), since their discovery (Iijima, 1991), has been an optimum material for reinforcement of metal matrices, because of their excellent mechanical and physical properties (Xie, et al., 2000; Troiani, et al., 2003) as well as high thermal and electrical conductivity (Che, et al., 2000; Deng, et al., 2007; Chen, et al., 2017), as shown in many researches (Bakshi, 2010). Moreover, every other process is already more expensive and sophisticated for industry applications.

There are many processes for incorporating carbon nanotubes in metal matrices (Bakshi, 2010) and, among them, powder metallurgy is widely used, because of their versatility and economy. For example, recently, Fattahi et al. (2013) used this process for aluminum metal matrix in incorporation via sintering of addition metal and subsequent arc welding with good results. Besides, homogeneity dispersion and agglomerations, promoted by the Van der Waals forces, are considered the main problems to solve (Chen, et al., 2015).

In this work, different times of carbon nanotubes dispersion in austenitic stainless-steel metal matrix by mechanical alloying were studied. XRD performed crystalline structure analysis, Raman spectroscopy was used to determine CNT defects and Scanning Electronic Microscope (SEM) performed the characterization of dispersion and homogeneity.

2. BACKGROUND AND THEORETICAL CONCEPTS OF THE CARBON NANOTUBES

2.1 Discovery and properties

Until the 1980s, diamond and graphite were the only known crystalline forms of carbon. However, the various carbon hybridizations were already known. This led to the search for new carbon structures. In 1985, the England chemist Sir Harold Walter Kroto of the University of Sussex, in collaboration with Robert Curl and Richard Smalley of the University of Rice (USA), research on the discovery of a new allotropic carbon: the fullerenes (for example, the best known of them, the C₆₀, whose structure resembles a soccer ball containing 60 carbon atoms arranged in 20 hexagonal rings and 12 pentagonal rings (Kroto, 1985).

In 1990, during a symposium of the Department of Energy in Washington D.C, Smalley and Dresselhaus discussed the possibility that fullerenes had elongated structures forming tubes. In the same year, Dresselhaus discussed the electronic and structural properties of these fullerene longitudes that would be called carbon nanotubes. A year later, the fourth form of allotropic carbon was discovered by the Japanese scientist Sumio Iijima, studying carbon fibers synthesized by the technique of arc discharge (Iijima, 1991).

Subsequent analyzes have shown that carbon nanotubes are formed by graphene sheets rolled in the form of cylinders, where carbon atoms form covalent bond (strong) made by sp² hybrid orbitals. Carbon nanotubes form tubular structures with a diameter of less than 1nm or even tens of nanometers and lengths ranging from several micrometers to a fraction of a millimeter. The nanotubes are called single wall nanotubes (SWNT), Figure 1, when a single layer forms them or multiple wall nanotubes (MWNT) when concentric layers form them.

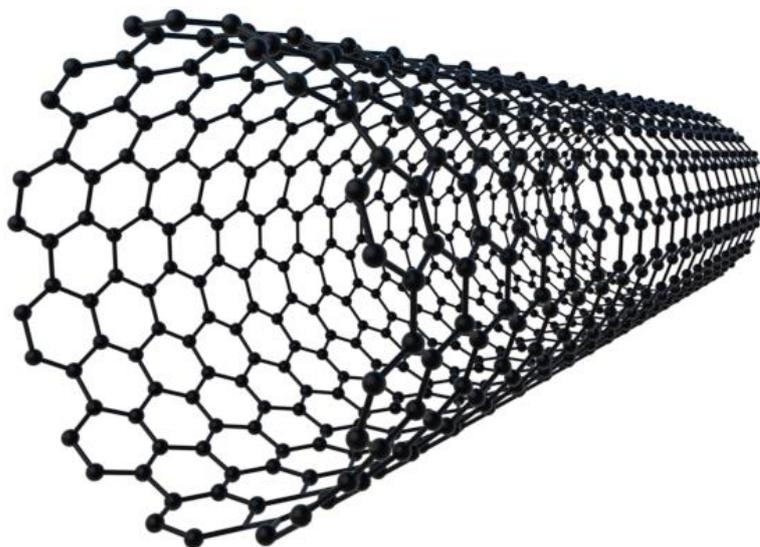


Figure 1. The above image is from a Single Wall Carbon Nanotube (SWNT). Single-walled carbon nanotubes (SWNTs) are nanometer-diameter cylinders consisting of a single graphene sheet wrapped up to form a tube.

In addition, carbon nanotubes are nanostructured materials that have special properties that depend only on the diameter and chirality of the tube. Carbon nanotubes can be semiconducting or metallic, this depends on the chiral vector, that is, the way the hexagons are oriented in relation to the main axis of the tube. Depending on how the crystal structure of single wall nanotubes is arranged, that is, their chirality, the nanotubes can have different mechanical and electrical properties. The chiral vector, given by the angle θ in relation to the structure is called zigzag when the angle is 0° and forms the 'armchair structure' when diverge 30° . Any other form with a different angle is called chiral (Pradeep, 2007).

Moreover, due to the bonds between carbon atoms, CNTs have a tensile rupture strength greater than Kevlar and steel, with the tensile strength being approximately 100GPa (Eatemadi, et al., 2014). The nanotubes have a great ability to withstand large deformations, up to 40% of their size, can recover of serious structural distortions, and have great resilience due to the graphite sheet (because the carbon atoms are capable of re-hybridizing) (Pradeep, 2007). Figure 2 use comparatives elements to explain why carbon nanotubes assume an extremely interesting position for the industry, being able to be incorporated into several metallic and polymer matrices in order to increase their degree of resistance.

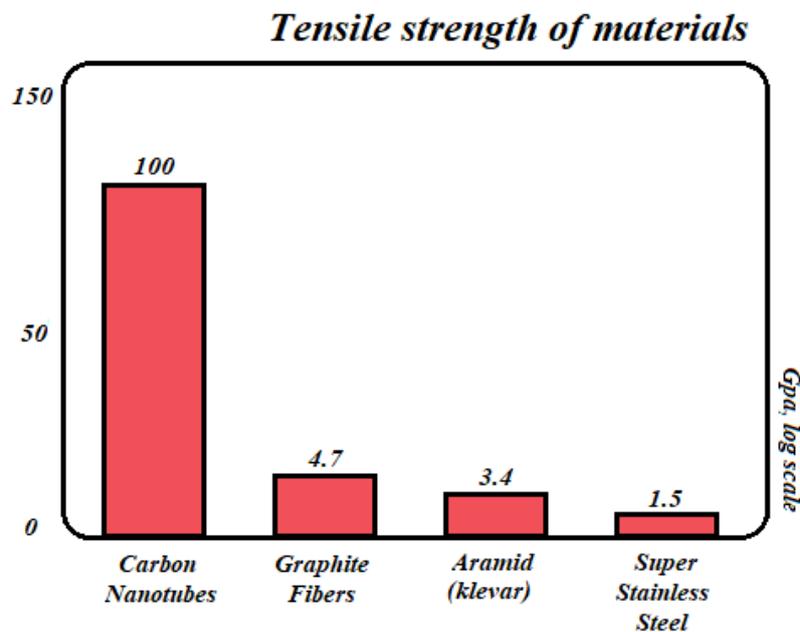


Figure 2. Comparative image of different materials to Carbon Nanotubes. For example, the comparison with Super stainless steels, such as ballistic steel, because this material have the best and the highest resistance in the market, and its tensile strength is up to 3x higher than of a ordinary steel. There is also Kevlar, the aramid-based polymer whose strength exceeds that of steel by up to five times per unit weight.

2.2 Synthesis methods

In the first processes to synthetize carbon nanotubes the used techniques were laser ablation and arc-discharge. Both techniques can produce both single and multiple-walled nanotubes and it stills continue to be used today. Laser ablation is not capable of producing large-scale nanotubes, on the other hand, the arc-discharge method produces, but at the same time, a large amount of carbon and amorphous carbon structures are produced which require a purification phase to obtain the nanotubes. More recently, the method of chemical vapor deposition (CVD) has been widely used in the growth of NTCs since it allows better control of nanotubes at lower temperatures compared to other processes. It is worth mentioning that one of the reasons why carbon nanotubes are not yet used commercially and intensively these days is due to the fact that it is not yet possible to produce nanotubes in a large scale in a purified and affordable way, although large progress has been made recently (Hata, et al., 2004).

2.3 Mixing processes

Since the beginning of the 2000s, due to the excellent properties of carbon nanotubes, the research to use them as reinforcement in polymeric, ceramic and metallic materials was highlighted to the scientific community. But regarding the nanotube aggregation process in metallic matrices, some difficulties must be emphasized, such as the homogeneous dispersion of nanotubes in the matrix, the state of aggregation of NTCs and their chemical and structural stability, problems that can be solved by applying methods wherewith the mixture occurs with the decrease of these unwanted effects (Bakshi, 2010).

It should also be noted that the nanotubes are very flexible materials, do not suffer damage in their structuring when folded or subjected to great pressures; when it happens in multiple wall nanotubes, the more outer layers tend to break, and if they have a defect in their structure, their resistance will be less (Sales, 2013). In addition, the mixture system adopted to add carbon nanotubes in metallic stainless-steel matrix should cause the least possible damage to the nanotube. Because of this, some different ways are utilized to incorporate the carbon nanotubes in metal matrix, as show in the Figure 3.

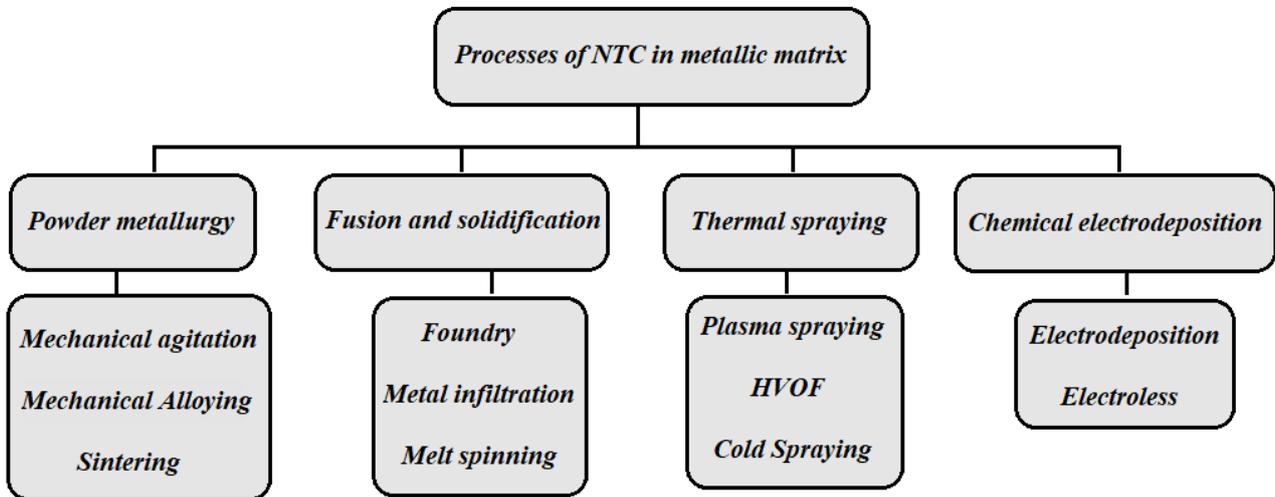


Figure 3. Schematic representation of the different methods of synthesis of carbon nanotubes in metal matrix.

For the preparation of the powder, as the initial step of the process, the Mechanical Synthesis method deserves attention due to its practicality, low cost, and mainly because the matter remains in the solid state all the time, which prevents possible limitations that phase diagrams may present in different methods (Campos, 2005).

2.4 Mechanical Alloying

The Mechanical Alloying process consists of processing powders through repeated cold welds, fractures and re-soldering of the particles in a high-energy mill (Campos, 2005). The method is performed through a ball mill, which is filled with the powder of the metal matrix, the compound wherewith it is desired to aggregate and rigid balls, being rotated with a certain speed (Suarez, Reinert, Mucklich, 2016). These processes were represented on the figure 4.

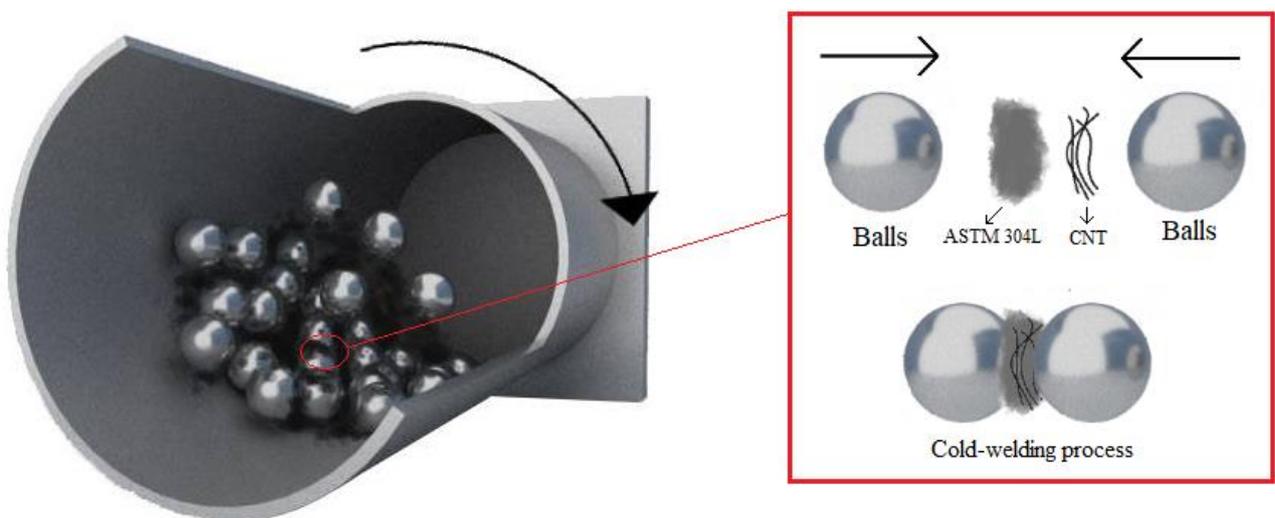


Figure 4. Schematic representation of the Mechanical Synthesis process: the mill with the compounds being struck, and thus, cold-welded by the spheres. Cold-welding is a process of joining materials, without the use of heat sources, which maintains its physical, chemical and mechanical characteristics (Off-scale image).

The process, although simple, presents some variables that can interfere radically in the process, being able to be independent or not. Among them, the following stand out (Campos, 2005):

- The types of mill, whose main models are those of agitation, which works with few sample amounts (10g); Those of friction, that work with more significant amounts (from 500g to 4Kg); Or planetary mills, that can contain more than one container at a time, if less than 250g each;
- The material used in the mill, due to the friction to which it is submitted, can be contaminated by the material that can detach from the wall of the container, contaminating the sample or changing the chemical composition of the powder;
- The rotation speed, both by the limitation of the mill to be used, and by possible damage to the sample due to the repeated impact of the spheres container;
- The milling time, which must be calculated to allow breaks between the fracture and the cold-welding of the powder particles. As well as the rotation speed, the grinding time should be minutely controlled to avoid damaging the sample.

2.5 Characterization

The characterization of the mixture can be performed by: using X-ray diffraction to analyze the crystalline structure of the materials; also by Raman Spectroscopy, which is used to analyze the degree of defects of the nanotubes and with the utilization of SEM to obtain images with which the characterization of the dispersion state and of the best nanotubes aggregation in the metallic matrix. Photoluminescence spectroscopy and TEM (Transmission electronic microscopy) are also widely used to perform the characterization of the carbon nanotubes.

3. MATERIALS AND METHODS

The multilayer carbon nanotubes were produced by the **CTNanotubos** via chemical vapor deposition (CVD), based at the Federal University of Minas Gerais, and the AISI 304L steel powder, grain size of 44 μ m, was acquired by **BRATS Company**. The CNTs were mixed with the stainless-steel metal matrix at a 5% by weight ratio, to assure that, even with possible material losses, there would still be enough nanotubes do be characterized.

Then, 40 ml solution of isopropanol (Reis et al., 2016) was added to the mixture (Fonseca, 2012), with the aim of evaporating the most of carbon impurities that are not interesting in the process (Silva, 2006) generated by the remaining metallic catalyst (Ferreira, 2004), and to break the Van der Waals forces that connect the sheets, which may hamper CNT aggregation to the metal matrix (Chen et al., 2015). Subsequently, the sample was dried and weighed.

After drying, the mechanical alloying process was performed. The sample was inserted into a planetary ball mill, together with 25 stainless steel balls, 1cm in diameter each, being coupled to a lathe for a rotation of 200 RPM (Fonseca, 2012). Teflon tape was used to seal the mill to prevent any leaks from the material. To avoid possible overheating of the sample (Chen et al, 2015), there were 10-minute stop intervals every 10 minutes of synthesis. During a stage of MA, in addition to collecting a sample before the beginning of the process, samples were collected with 30 min, 60 min and 90 min of mixing. Among the possibilities found in the literature, the powder metallurgy process, using the mechanical alloying method, stands out due to the success of adding CNT to metal matrices such as aluminum (Esawi et al., 2007; Fonseca, 2012; Fattahi et al., 2013), Copper (Kim, et al., 2006) and Silicon (Eom, et al., 2006). The respective processes were represented Figure 4.

To evaluate aggregation state of the carbon nanotubes in the metal matrix was used a Jobin Ivon, model T64000 Raman spectroscopy equipment, conducted with a wavelength of 532 nm. Besides, the peaks characteristic of the CNTs and SS 304L were analyzed by X-Ray diffract Bruker, model 8 Advance with Bragg-Brentane geometric, Lynx Eye detector, Cu tuber, Cu radiation ($K\alpha_1=1.540598 \text{ \AA}$) and Ni filter $K\beta$. At last, Scanning Electronic Microscope VEGA3 TESCAN characterized the different times of milling. The process was represented in figure 5. All this process was already developed over patent deposited.

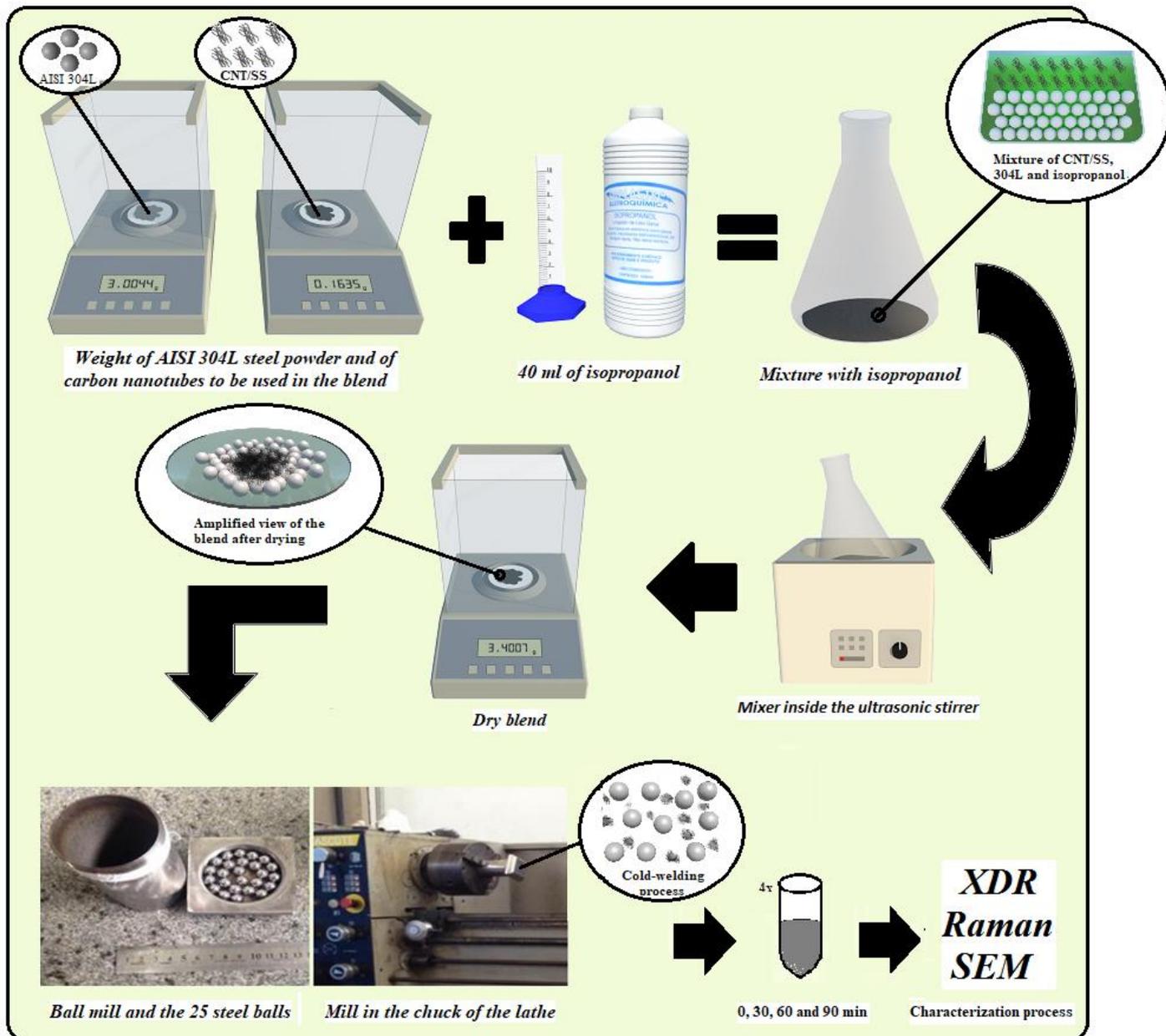


Figure 5. Schematic illustration of dispersion a mixing of CNT/SS 304L. In the scheme, extensions were made in order to show the process on a microscopic scale.

4. RESULTS AND DISCUSSIONS

4.1 X-ray Diffraction

Recently, Bera et al. (2006) used the XRD method to characterize a sample of nanotubes and found that the characteristic spectrum of the material occurs in the reflections (002) and (004) in the angle between 25.4° and 26.4° (Chen et al., 1999; Bera et al., 2006). Which can be identified as a hexagonal annular structure of the graphene sheets that MWCNTs are formed. Figure 6 compare diffraction analyzes of the pure nanotube samples and the mixture obtained in the mixing at 60 minutes. From the analysis of the graphs was observe that the first presents the characteristic peaks of the CNTs with the spectrum of nanotubes at the point (002), and the mixtures present both the peaks of the CNTs and the steel AISI 304L, which shows austenite in the points (111), (200), and (222), and martensite at the (110) point (Jain, Christman, 1996). In addition, the characteristic curve of the CNTs becomes smoother as the mechanical synthesis time is increased, reaching almost no curve highlighting in the 60 min sample. This can be due to several factors, such as the increasing amorphization of nanotubes, because with a longer mechanical alloying time the damage of carbon nanotubes increase, and a growing state of dispersion of CNTs in the metal matrix (Esawi, et al., 2009).

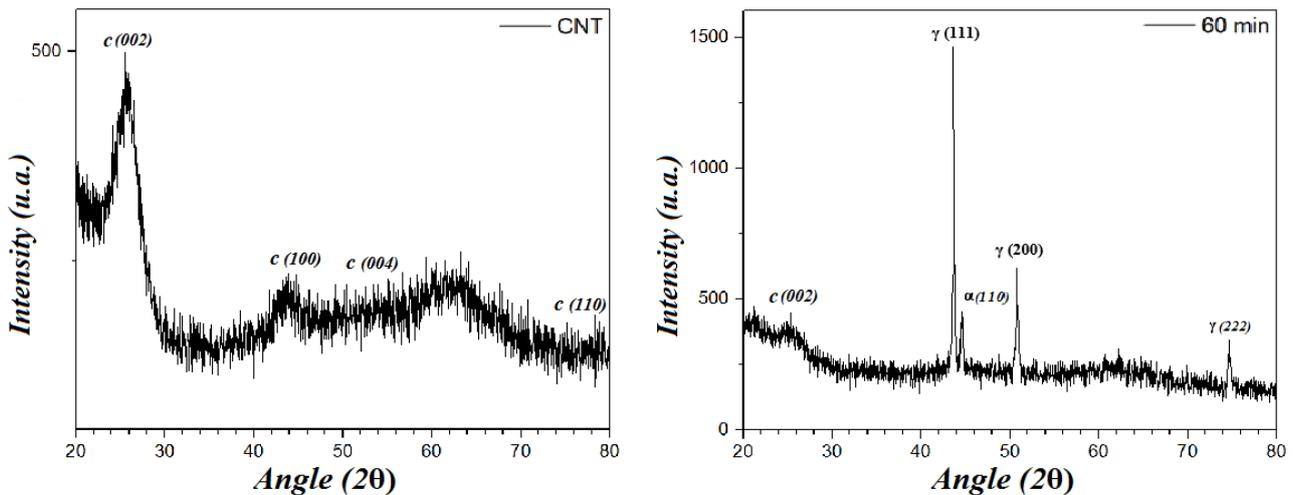


Figure 6. The images show the XDR results of the pure carbon nanotube samples and the sample obtained after 60 min of Mechanical Alloying. The peaks C (002), C (100), C (004) and C (110) are the representations of MWCNT characteristic planes, which occur approximately at angles 26°, 43°, 54° and 78°. This behavior is characteristic of nanotubes and repeats in the samples that passed through MA, which proves the aggregation of the nanotubes to the metallic matrix, the other peaks of the sample of 60 min represent the characteristic components of the steel: austenite in the points (111), (200), and (222), and martensite at the point (110).

4.2 Raman Spectroscopy

The micro-Raman spectrometer was used to measure the damage degree of the nanotubes inserted in the mixture, so the time of mechanical synthesis that less damaged the material can be determined, as display in the figure 7. Samples were submitted to two analyzes at different points (D and G) so that a comparison could be made. The spectra referring to the D and G bands, characteristic of the standard behavior of the Carbon Nanotubes (Bokobza, Zhang, 2012), are present in all analyzed samples.

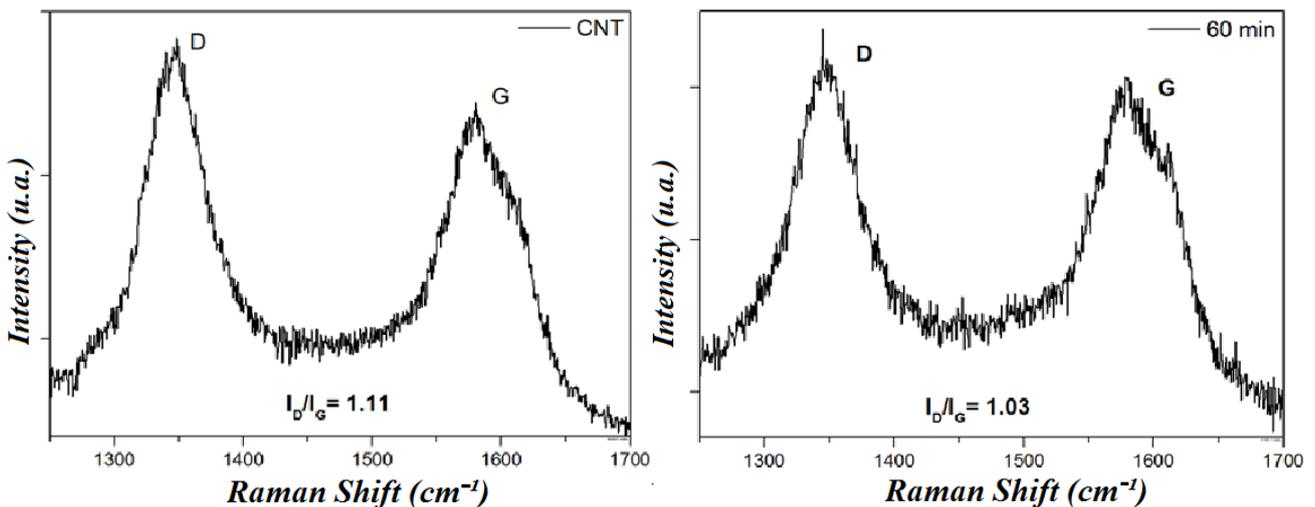


Figure 7. The factors that generate this behavior of nanotubes for Raman spectrometry are quite complex. Therefore, it is up to us to analyze the permanence of the behavior of the graph, thus verifying the degree of defects introduced to the nanotubes by the MA process. The graph shows the comparison of the behavior of the G and D bands of the pure nanotubes and of the mixture at 60 min. It is noticed that the 60 min mix lightly altered the graphical behavior, suggesting a lower generation of defects during the synthesis processor and consequently a more adequate mixing.

Fig. 7 show peaks D and G in 1347 and 1584.6, respectively, for CNT. Furthermore, peaks D in 1346.2 and G in 1583 were observed with 304L plus 5 wt.% CNT after 60 min of Mechanical Alloying. These results were found in other researches about CNT incorporation in metal matrix such as aluminum (X. Zhu, et al., 2016) and magnesium (G. Q. Han, et al., 2016). Moreover, figure 7 show the I_D/I_G intensity ratio, used for measured the defects level. It calculated

by Gaussian distributed analysis as for CNT as received and SS 304L plus 5 wt.% CNT, these rates were 1.11 and 1.03, respectively. Whence, no additional defects were found and even this level was minor after 60 min of MA, which could mean that CNTs might be better aggregated to the metal matrix.

Table 1. Center and intensity of Raman spectra peaks D and G

	D		G		I _D /I _G
	Center	Height	Center	Height	
Carbon Nanotubes	1347	1880	1584.6	1688	1.11
60 min SS 304 L + CNT	1346.2	2894	1583	2816	1.03

4.3 Scanning Electron Microscopy

Fig. 8 (a) and (b) shows carbon nanotubes and austenitic stainless steel AISI 304L as received, respectively. Figure 8 (c) display CNTs and SS 304L in 0 min, several regions of carbon nanotubes and stainless steel are observed separately. After 30 min of MA, the nanotubes were added to the metal matrix, as observed in the figure 8 (d), however, there are a lot of CNT clusters and agglomerations of nanotubes. It can be verified that in several regions of the stainless steel grain there is the presence of nanotubes dispersed in the metal matrix, which indicates that the homogenization process is already beginning to take place. However, CNTs are still heterogeneously aggregated, which is not interesting for a dispersion optimization. Figure 8 (d) illustrates the state of aggregation of the mixed carbon nanotubes to the stainless steel powder, visualizing how they dispersed in the metallic matrix, and compare with the results obtained by Raman Spectroscopy and X-ray Diffraction. As can be observed in the figure 8 (e), after 60 minutes of MA, it can be verified that the nanotubes were better aggregated to the metal matrix. In addition, CNTs dispersed more evenly throughout the mixture, ensuring that by examining random areas in the sample, the presence of nanotubes attached to the stainless steel powder could be verified which was not possible with less time of synthesis. For the time of 90 min, the CNTs are beginning to be destroyed and a heterogeneous dispersion is observed again, it is show in the figure 8 (f).

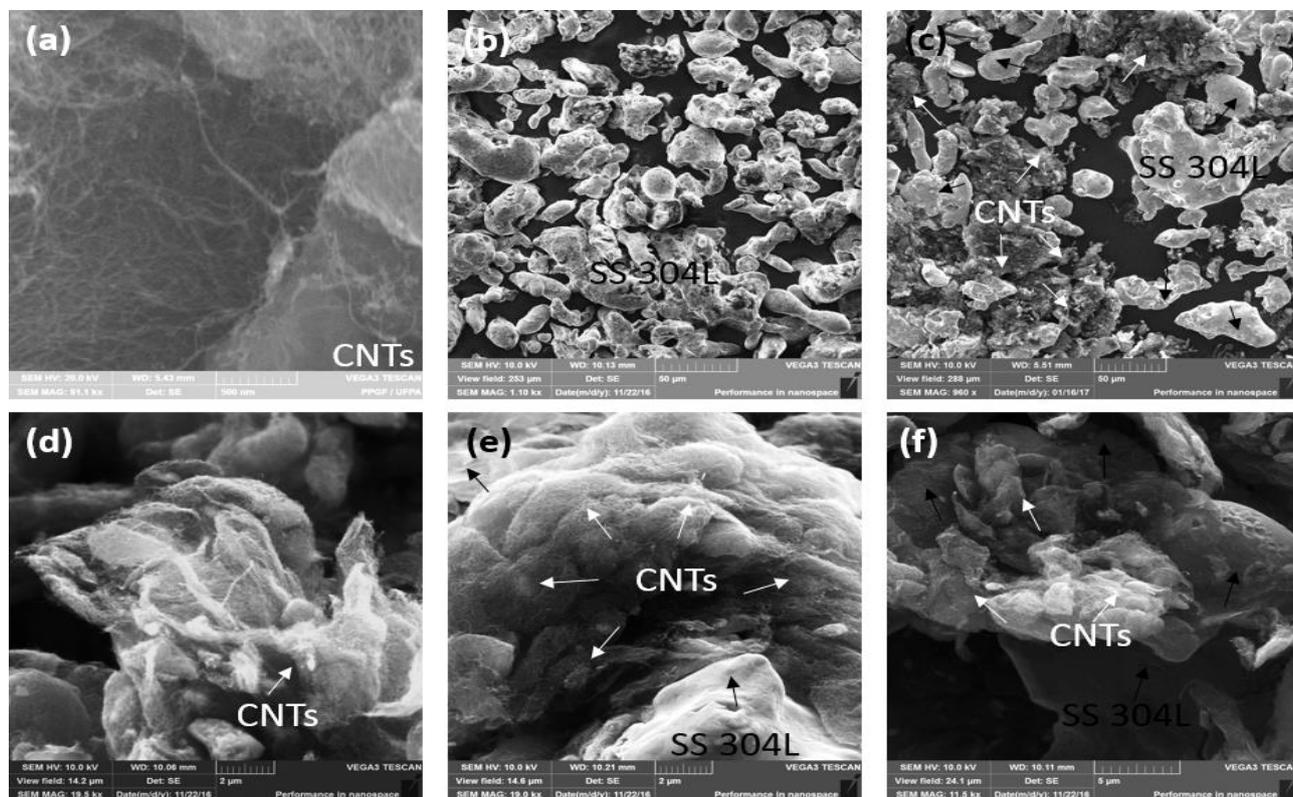


Figure 8. (a) Carbon nanotubes as received, (b) Austenitic Stainless Steel (SS) 304L Powder as received (c) SS 304 L plus 5 wt.% CNTs mixing through of mechanical alloying (MA), at 0 min, (d) 30 min, (e) 60 min and (f) 90 min.

Fig. 9 shows more clearly the state of aggregation of the nanotubes to the metal matrix after 60 min of MA. The successive expansions of Fig. 9 (a) and 9 (d) show images of different points of this sample, these images show greater homogeneity in the dispersion of the nanotubes in the steel and, consequently, a better aggregation of the CNTs to the matrix compared to previous samples. The analysis of points 9 (c) and 9 (e) shows that when observing different points of the 60 min sample, it is possible to find the presence of the CNTs, unlike what occurred with the samples of figure 7 (c), 7 (d), 7 (e) and 7 (f). This better dispersion of the CNTs in steel will culminated in better aggregation of the desired characteristics of the nanotubes to the steel.

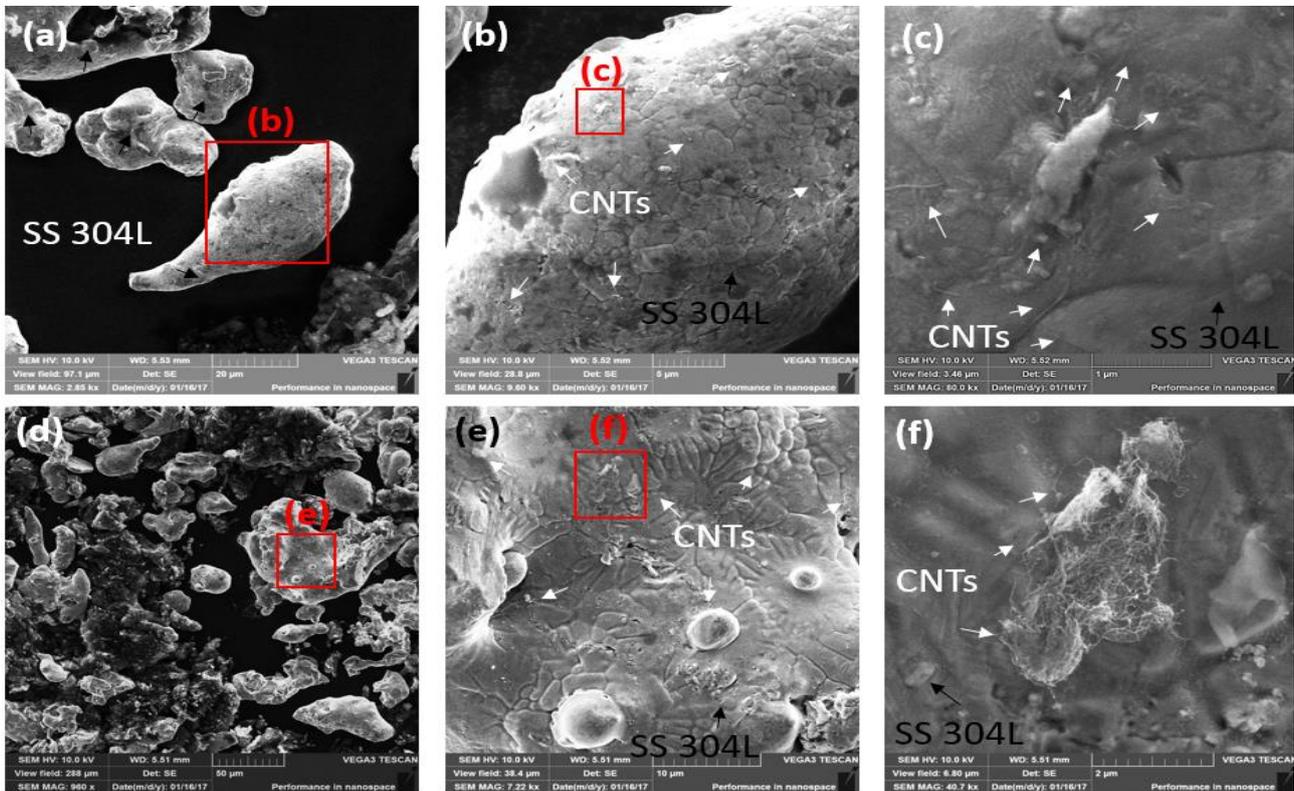


Figure 9. MA with 60 min in two different zones (a) and (d) of the powder mixture (a) 20 μm , (b) 5 μm , (c) 1 μm , (d) 50 μm , (e) 10 μm and (f) 2 μm . This image proves that the incorporation was better in this time.

5. CONCLUSIONS

Carbon nanotubes were successfully aggregated in ASS 304L via mechanical alloying, determining an optimal dispersion time in a planetary ball milling for this metal matrix. XRD and Raman Spectrometry both allowed to determining which parameters were most fit to produce the mixture with less defects; alongside SEM images, the mechanical alloying period that presented the best aggregation and dispersion was in 60 min.

6. ACKNOWLEDGEMENTS

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