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X-ray Tomography, AFM and Nanoindentation Measurements for Recyclability Analysis of 316L Powders in 3D Printing Process

Nima E. Gorji,*, Robert O'Connora, Dermot Brabazon

I-Form Advanced Manufacturing Research Centre, Collins Ave. Dublin 9, D09 W6Y4, Ireland

Corresponding Author: Nima.Gorji@dcu.ie

Abstract: Recyclability of the leftover metallic powder within the Additive manufacturing (3D printing) process has been less systematically investigated by the research groups although it is a usual practice in most academic and industrial laboratories to reuse the leftover powders for subsequent printing cycles. A better understanding of these mechanisms will assist in optimizing the number of times the recycled powder can be reused in the process to reduce the powder waste. We have recently focused on characterization of recycled powders left in the powder bed after the powder bed fusion process and evaluated the extent of porosity in the powder particles. X-ray computing tomography technique (XCT) has been used to analyze the concentration of porosity, inclusions and dendrites induced inside the recycled powder particles and compared that to the fresh counterparts. The XCT resolution of 2 μm was set to separately scan the powder badges for 3 hours. A roughly 10% more porosity has been calculated in reused powder particles (in at least 10 times reused power). Atomic Force Microscopy (AFM) was used to measure the roughness of the surface of powder particles which shows average roughness of 4.29 nm and 5.49 nm for the virgin and recycled powders, respectively. Nanoindentation measurement were also applied on a number of locations of the particles to compare the hardness of the virgin and recycled powders. For example, the recycled powder shows smaller a hardness of 207 GPa and an effective modulus of 9.60 GPa (average values) compared to 237 GPa and 9.87 GPa (average values) for it's virgin counterpart which can be correlated to porosities created beneath the surface. Nanoindentation was also applied on (micro and nano) polished surface of the particles under a force of 250 μN for up to 10 seconds. The stainless Steel 316L powder has been the material under study with the powder particles of average size 50 μm which were analyzed using Xradia XCT, Bruker Dimension ICON AFM and Bruker HYSITRON TI Nanoindentation systems. Further investigation is ongoing to correlate the mechanical properties of the manufactured parts to the microstructure and chemical compositions of the virgin and recycled powders.

Keywords: Additive Manufacturing; 3D Printing; Powders Recycling; AFM; X-ray Computing Tomography; Nanoindentation.

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1. Introduction

The recyclability of metallic powders within additive manufacturing (AM) process has been recently attracted the attention of many research groups and industries. Recycling the leftover powder during the powder bed fusion process has several advantages for driving the AM process to a greener and cost-effective technology. In order to reuse the recycled powders in the secondary manufacturing cycles, a thorough characterization is essential to monitor the surface quality and microstructure variation of the powders affected by the laser heat within the 3D printer. Most powders are at risk of surface oxidation, clustering and porosity formation during the AM process and it's environment [1,2]. Our latest analysis confirms the oxidation and the population of porous particles increase in recycled powders as the major risky changes in stainless steel 316L powder [3,4]. There is not much reported in the literature on treatments to bring the quality of the recycled powders to its initial stage for subsequent reuses. Sieving is a routine treatment before the reuse of recycled powders. However, that cannot reduce the surface oxidation or porosity of the particles which can be the source of porosity formation in the parts and ess laser absorption in the metling pool. Therefore, the best advice is to adjust the manufacturing process such a way that the recycled powders can be least affected during the process and thus not much of treatment is required in the subsequent cycles. Mixing the recycled powder with fresh feedstock is also a routine method to level up the mechanical properties of the manufactured parts [5,6]. It must be noted that many risk intolerant applications (such as biomedical and aviation industries) do not use the recycled powder at all because any abnormality in manufactured parts (sourced to recycled powders) can be significantly costly and unsafe. A part made of the recycled powder must have comparable mechanical properties to the one totally made of the fresh powder. For example, the hardness and effective modulus of these secondary parts must be in a reasonable range and comparable to the virgin-powder printed parts. Therefore, characterization of recycled powders can shed light on our understanding of powder quality for reuse in the AM process. It is known that the part's microstructure will have a direct relation to the condition of the virgin powder. Therefore, the subsequent use of recycled powder will alter the mechanical strength of the part, for example, by higher oxygen absorption on the surface of the powder. Oxidation can unfavorably impact on powder melting rate under laser heat and thus the released oxygen is absorbed by the powder particles near the melting pool or the released oxygen can remain beneath the melted area and cause porosity after solidification. The increased oxygen content can adversely affect the remelting of the particles and finally on the microstructure of the final part.

Here, we report our latest effort to measure the distribution of porosity formed in the recycled powders using the X-ray computing technique and correlate those analyses to the mechanical properties of the powders (hardness and effective modulus) obtained through AFM roughness measurements and nanoindentation technique.

2. Materials and Experiments

The material used for 3D printing was Stainless Steel 316L powder with average of 50 μm particle sizes. The chemical composition is O, C, Mn, Fe, Si, Sb as described in our previous paper [3]. 9 test cubes of 5 mm × 5 mm × 5 mm were printed using EOSINT M280 SLM 3D printer (powder bed fusion process) with 195 W power, 1000 - 1200 mm/s scan velocity, 0.1% limit on O2 in AM chamber, 80 °C ambient temperature and 0.1 mm beam diameter. The

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recycled powder was removed from the powder bed by vacuum, sieved for particles or spatters of bigger than 50 μm and been reused for 9 times in the machine. Then again sample powders were collected from the powder bed after the print completed and were labeled as recycled powders.

Both virgin and recycled powders were analyzed by number of techniques including XCT and Nanoindentation. XCT was performed by X-ray computed tomography (XCT) measurements were performed with a Xradia 500 Versa X-ray microscope with 80 KV, 7 W accelerating voltage and 2 μm threshold for 3D scan.

To measure the roughness of the virgin and recycled powder particles, we performed Atomic Force Microscopy (AFM) and confocal microscopy using the Bruker Dimension ICON AFM. The average roughness was calculated using the Gwyddion software to remove the noise and applying the Median Filter on the images as a non-linear digital filtering technique.

The nanoindentation on several powder particles were also run using a Bruker HYSITRON TI Premier. The particles were cold mounted on mould silicon and polished with Metkon Forcipol polisher using MetPrep Tri Hard solutions (Diamond and Silicon) for nano and micro polishing. The pore areas on the powders were identified by optical microscope and the nanoindentation was performed on several locations on the powder to find the impact of porosity on the hardness and effective modulus of the recycled powders.

3. Measurements and Characterization

The XCT images were analyzed using Aviso and ImageJ softwares available to the system. Figure 1a shows the 3D image reconstructed from all the 900 CT slices recorded by the camera. The particles are visible in 3D format. From this image, we select a region of interest as shown in Fig 1b and apply image processing methods to extract the pore size and distribution inside the particles. A slice with internal porosities has been displayed in Fig. 1c with red arrow as an example. Fig. 1d presented the captured pores in this slice after applying the image processing. The metallic oxide elements are more prone to oxidation such as Mn and Si and this has been more occurred in our AM process. These elements have a high affinity to oxygen thus the formation of such metallic oxide product is hardly avoidable. The chemical composition of 316L (from powder manufacturer) is Si (0.5%), Mn (1.8%), Cr (16.7%), Mo(2%), Ni (10%), Fe (Bal.) [7]. Stainless Steel 316 of our prevous study has shown to form metallic oxides of MnO, CrO and FeO on it’s surface under laser heat [5].

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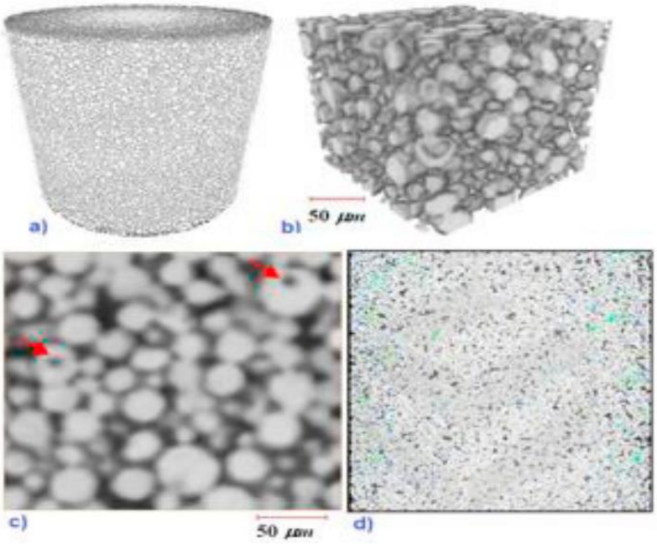
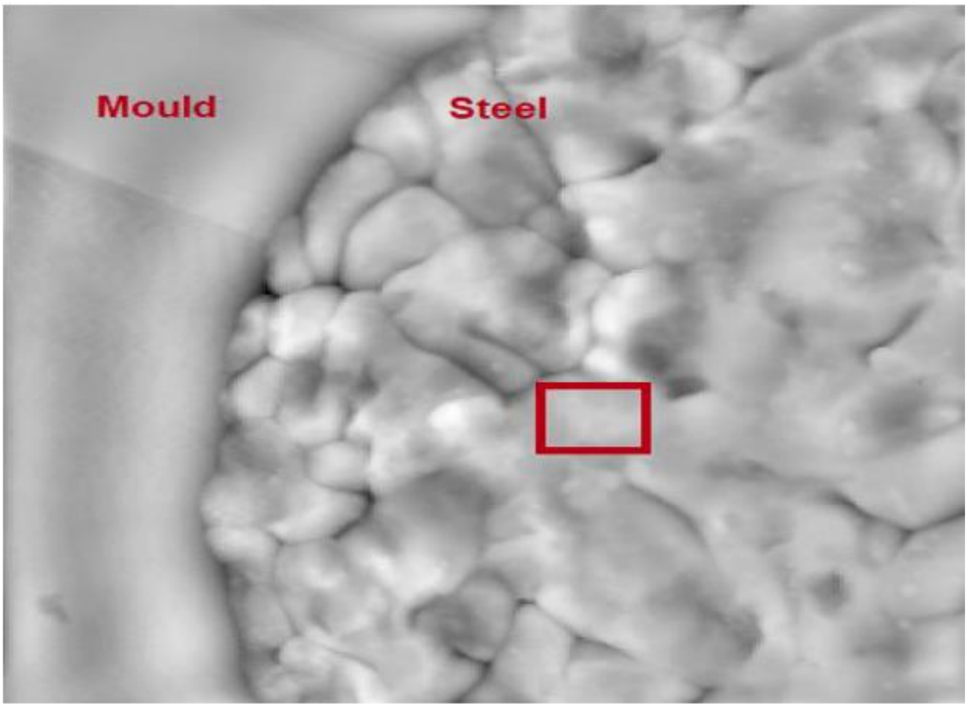


Fig. 1. XCT imaging of the powder. (a) 3D rendered image of all 900 recorded CT images, (b) selected region of interest, (c) internal pores in particles indicated in a 2D slice, (d) identified pores inside the particles after the image processing method.

The AFM topography images were processed by Gwyddion software which allows dragging a straight line across a single grain and calculates the average surface roughness. Fig. 2 shows the topography of a particle surface where the mould and steel can be clearly distinguished. A straight line was horizontally placed in the central area on the steel (as shown by square) to measure the surface roughness. This was repeated for both virgin and recycled powders.



Nanoindentation was also applied on the particles in different locations with force of 250 μN kept for up to 10 seconds. Fig. 3a shows the prepared sample of particles sparkled on the cold

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mount and polished in nano/micro size. Fig. 3b shows a typical indent applied on the surface of a recycled powder particle.

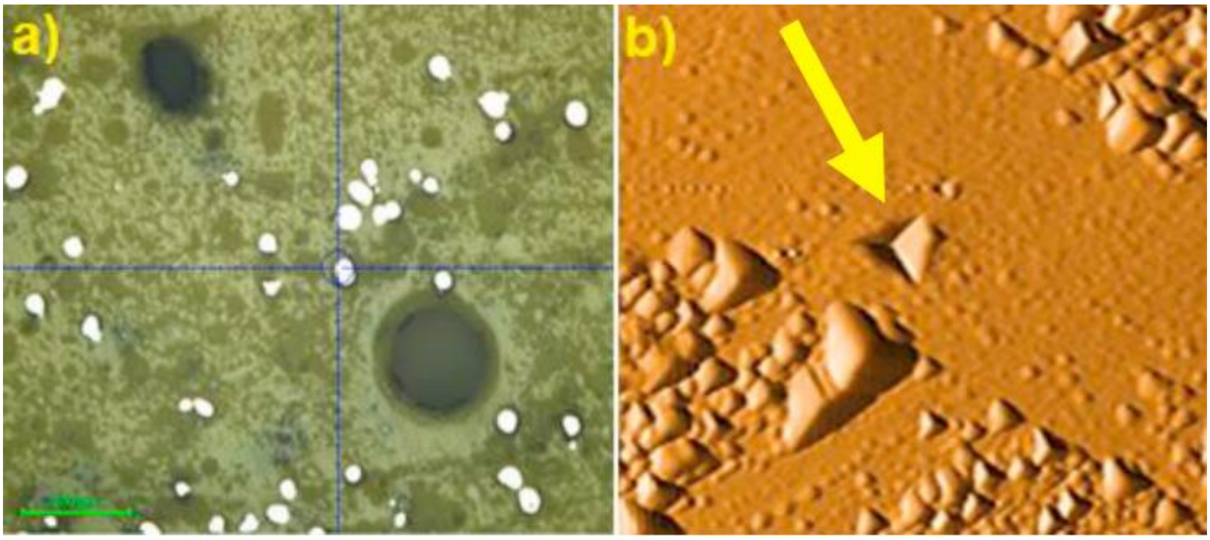

Fig. 3. (a) powder particles placed on hardening mold for nanoindentation, and (b) an indent applied on a particle surface.

4. Results and Discussion

The characterization of the virgin and recycled powders by XCT and nanoindentation has been presented in Figs. 4 and 5. Fig. 4 shows the variation in population and size distribution of the pore formed inside the particles. The pore size in recycled powders has a wider distribution compared to virgin counterpart. The main population of pore size is around 1-5 μm in virgin powder which slightly reduces to bigger size but for a smaller population. There are also bigger pores in recycled powder but with a smaller population. On the other hand, looking at higher pore population in virgin powder (around 10 μm size), we believe that the out-diffusion of metallic elements to the surface occurs during laser irradiation. The out coming metallic elements oxidize on the surface due to their high oxygen affinity and, therefore, would not return into the bulk again thus a vacant hole remains inside the particle [2,3]. Another definition is that when reusing the recycled powder, the oxide layer on surface is melted under laser and the released oxygen remains in the bulk as a trapped gas and forms the pore [7]. According to dynamic diffusion rate, the size of the pore would be different which actually seems to be a slow process since the most pores have a small size in the recycled powders. Therefore, one can also conclude that the porosity can form with higher probability for shorter laser exposure times. Geenen et al have concluded that at long exposure time of 90 μs can allow the gases to skip the meltpool and the pore formation can be minimized [8,9].

Fig. 5 shows the surface roughness measured across a 10 μm line on the surface of virgin and recycled powder particles. The average roughness of 4.29 nm and 5.49 nm were calculated for virgin and recycled particle which means that 3D printing process may increase the surface roughness of the recycled particles. This in incline with literature reports in Refs. [3,10].

Fig. 6 presents the nanoindentation results realized for both virgin and recycled powder particles. The hardness of the recycled powder is smaller than the virgin counterpart, which could be attributed to higher pore density in recycled particles. Obviously, porosity makes the powder more vulnerable to the applied force resulted in smaller hardness.

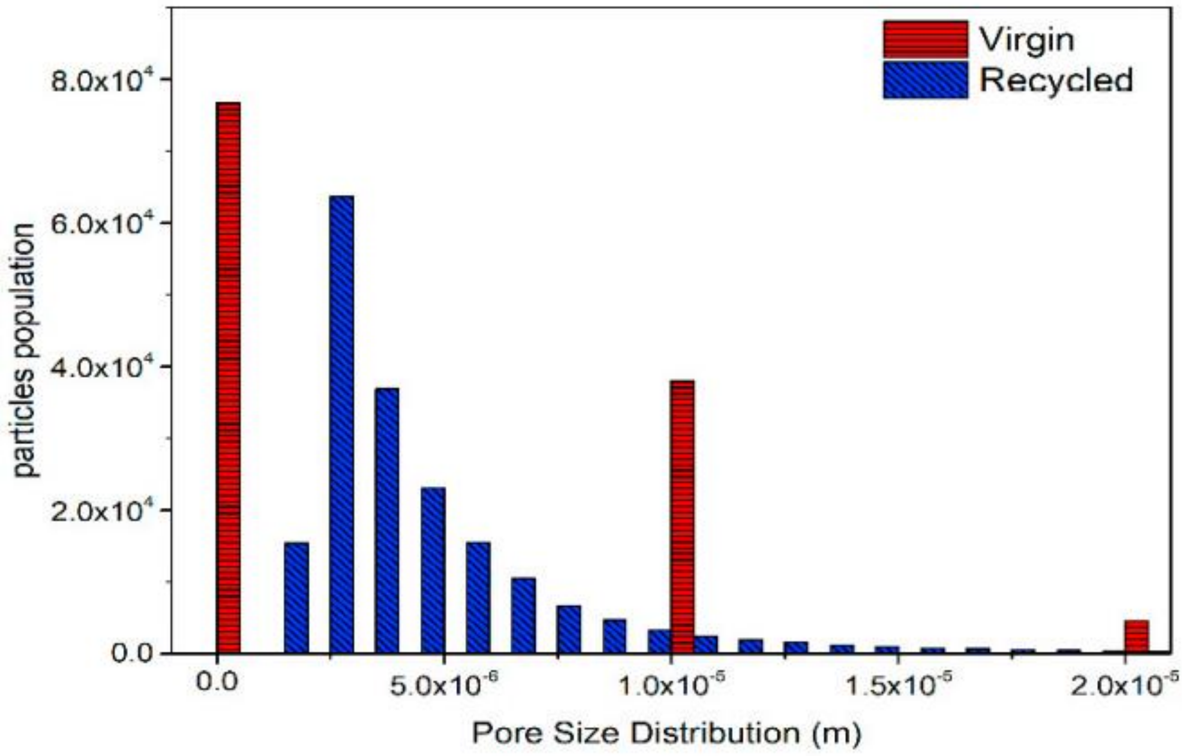

Fig. 4. pore size distribution in virgin and recycled powders extracted from image processing on XCT measurements.

Also the modulus is reducing for recycled powder for a range of the indents on different locations on the powder representing the impact of laser heat and the manufacturing process in developing pores and a softer bulk for the recycled powders. Geenen et al have also concluded that multiple reuse of the recycled powder can still have a more impact on its mechanical properties [11].

Alternatively, reduced mechanical properties can also be due to change in grain size of the powder particles as stated by Meier [12]. However, our SEM and AFM results have not detected much grain redistribution in the recycled powder (not shown here). Correlating the XCT and nanoindentation results shows that the higher porosity in powder reduces the hardness and modulus of the particles and that will damage the mechanical properties of the manufactured parts. Few methods are available to reduce the oxidation/impurities from the particle surface but the current practice of the users is sieving the bigger particles and mixing the recycled powder with the virgin in subsequent manufacturing cycles. Nevertheless, the porosity population and porosity formation may not be avoided by such simple powder post-treatments.

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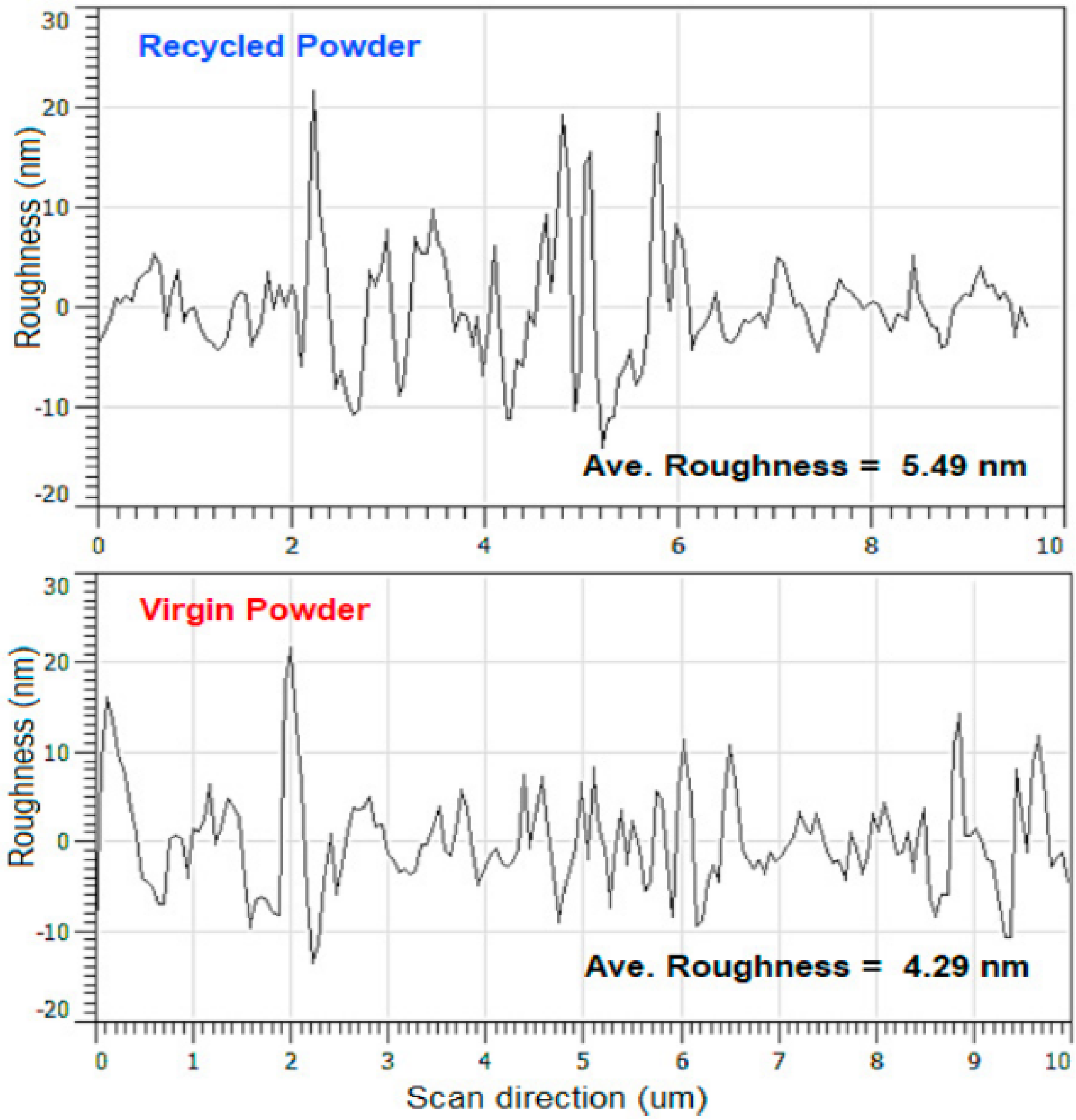

Fig. 5. surface roughness plots from AFM measuremetns on powder particles. The inset is average roughness calculated by Gwyiddion software.

5. Conclusions

We have performed XCT and Nanoindentation characterization on virgin and recycled stainless steel 316L powders used for powder bed fusion additive manufacturing process. The study aims to promote the mechanical properties of the parts printed from reused powders. this requires a clearer understanding of powder quality in terms of surface composition, pore population and size distribution. We have previously presented our achievement on surface and size analysis using SEM and XPS analysis. Here, we focused on pore distribution in both powders and correlated that to surface roughness, hardness and effective modulus obtained from nanoindentation analysis of the powder particles. The results indicate that pores population is about 10% more in recycled powders affected by the laser heat and oxygen inclusion/trap in the powder, which in turn, increases the surface roughness but reduces the hardness and modulus of the recycled powders. The pores are filled with gases (such as Argon or Oxygen) since these gases are not able to skip the melt and have a lower solubility in the melt throughout the solidification process. A rather systematic study is ongoing to correlate the tensile analysis of the parts manufactured from recycled powders to powder quality in terms of pore population and surface or bulk composition.

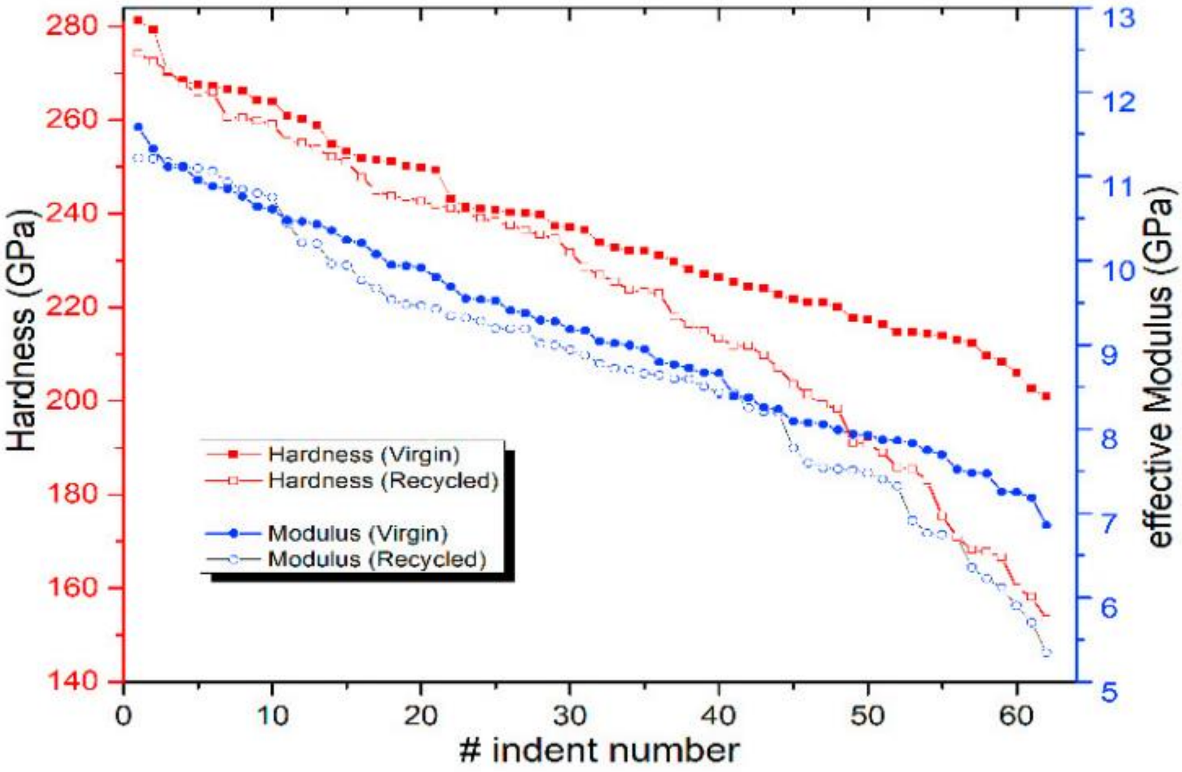

Fig. 6. Hardness and Effective modulus of the fresh and virgin particles by nanoindentation measurements.

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