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Effect of recycling on internal and external defects of Ti-6Al-4V powder particles for electron beam melting process

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Abstract

Electron Beam Melting is an additive manufacturing process that allows the creation of high-resolution parts with complex shapes, starting from a powder feedstock and using an electron beam as the energy input. Due to the high costs of the powder, the excess one must be reused otherwise a high percentage of unused powder would be lost after the printing process. The virgin and reused powders may have not the same characteristics, and therefore the printed parts may not have the same final quality which is detrimental to mechanical performances. However, this topic still needs research, as the influence of recycling on powder characteristics is not fully understood. In this study, the internal defects, and their distribution in virgin and recycled Ti-6Al-4V powders particles, are investigated by means of an optical microscope. For the analysis of the surface morphology and the external characteristics, it was used the Scanning Electron Microscope (SEM). At the end, a commercial software ImageJ was used in order to provide some quantitative results on the internal porosity percentage. The results demonstrate the dependence on the powder atomization process, which is primarily responsible for the micro-porosities, due to the trapped gas in virgin powders, and the rough surface morphology, including the presence of various satellites. As the number of reuses increases, the quantity of satellites decreases, probably due to the partial melting on the surface, which consequently returns in rougher surfaces of the recycled powder particles. In addition, most of the internal porosities disappeared making the cross-sectional faces of the particles near fully dense.

Keywords: Additive Manufacturing; Ti-6Al-4V; Electron Beam Melting; powder recycling; internal porosity; powder surface morphology.

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1. Introduction

Adding material layer-by-layer, instead of removing it, which is the primary feature of Additive Manufacturing technologies, allows the creation of very complex shapes (Guo & Leu, 2013), including lattice structures (Bellini, Borrelli, Di Cocco, Franchitti, Iacoviello, & Sorrentino, 2021) where a low weight is required (Bellini, Borrelli, Di Cocco, Franchitti, Iacoviello, Mocanu, et al., 2021), with no need to use removal or further post-processes (Gibson et al., 2010). Electron Beam Melting (EBM) is an additive manufacturing process for metals, which is part of the Powder Bed Fusion (PBF) category. The process takes place in a vacuum chamber preheated and uses an electron beam as the energy input, therefore gas contaminations and the formation of heat cracks are avoided (Galati & Iuliano, 2018) (Loeber et al., 2011). The major disadvantage of the PBF processes is the occurrence of a large number of internal and external defects that lower the mechanical properties, such as the high surface roughness considered one of the main weaknesses (Boschetto et al., 2021). Although the quality of the final component depends on various factors such as process parameters, it also strongly depends on the quality of powder feedstock which in turn depends on the atomization process used (Bellini, Berto, et al., 2021) (Iebba et al., 2017). There are several atomization processes and each of them has its own characteristics. For example, the plasma rotating electrode preparation (PREP) is known to provide near fully-dense powder feedstock, which allows the realization of almost free of porosities parts, while the gas atomization process provides a worse situation from this point of view (Ng et al., 2009). In addition, the presence of satellites (tiny particles) attached to the surface of particles is a common thing in gas-atomized powders (Yusuf et al., 2020). On the other hand, the biggest benefit compared to previous technology is the possibility to reuse the powder excess once the printing cycle is finished (DebRoy et al., 2018), and therefore the feedstock costs are lowered. However, recycled powders do not have the same features as virgin ones and it is necessary to understand the differences to avoid performance degradations in printed parts. Although the topic still needs research, in the scientific literature there are already interesting observations and results. Starting from a powder with satellites, as the number of recycles increases, the powder is observed to improve its external characteristics. The satellites disappear completely (Emminghaus et al., 2021), or simply decrease (Carrion et al., 2019), depending on the number of reuses. This change is due to the temperature conditions that cause the melting of satellites on the surface of the particles (Popov et al., 2018), resulting in an increase in the surface roughness. In addition, the particle size distributions narrow (Emminghaus et al., 2021), (Strondl et al., 2015), (Carrion et al., 2019), probably because the smaller particles are raised inside the printing chamber and remain in the ambient atmosphere, thus these particles are not recycled (Seyda et al., 2012), or probably because the smaller particles adhere to larger ones and avoid being counted (Sutton et al., 2020).

Contrastant results regarding the flowability were found. Some authors (Strondl et al., 2015) showed a different behavior between Ti-6Al-4V and Inconel718 powder particles. For the first one the flowability decrease with increasing the recycling number, while for IN718 powder it is the exact opposite. This behavior might be because the powder recyclability is material dependent (Sutton et al., 2020), thus not all materials show the same trends. But this seems not to be the case, since (Emminghaus et al., 2021), who analyzed a Ti-6Al-4V batch of powder, found that the flowability increased after multiple recycles.

The recycling processes are also known to be detrimental to the appearance and increasing the number of defects, which is due to both mechanical and heating reasons, i.e. sieving procedure and the temperature conditions in the printing chamber. The sieving procedure is responsible for deformed and broken particles, while the temperature conditions, including the pre-heating, are responsible for metallization, bonded satellites, elongated particles, etc. (Popov et al., 2018). Other authors (Ahmed et al., 2020) confirmed the presence of more irregular particles in reused powders, which may affect the level of porosity in the printed parts.

The increase in oxygen can be attributed to exposure to the ambient atmosphere during sieving and/or removal of parts from the build chamber (Seyda et al., 2012), this is why as the number of recycles increases, oxygen also increases. Other reasons for the increase of the oxygen level are the contamination despite the presence of inert gases in the build chamber, and the oxygen which might already exist in the powder feedstock, due to the atomization process (Yusuf et al., 2020). The oxygen limit value of 0.2% defined by ASTM might be reached after only 12 reuses (Petrovic & Niñerola, 2015), or even after 11 recycles (Ghods et al., 2020).

Some characteristics tend not to be significantly influenced by the change in powder characteristics. For example, differences in shape are not visible in some works (Strondl et al., 2015), and neither the phase distribution (Ahmed et al., 2020).

However, the effect of powder recycling is quite unexplored, therefore the aim of this paper is to provide some more information on this topic. In this work, two batches of Ti-6Al-4V powder particles were analyzed both from the external and internal point of view using a Scanning Electron Microscope (SEM), an Optical Microscope and the commercial software ImageJ.

2. Experimental

The material analyzed in this study is Ti-6Al-4V (grade 5) powder particles, produced by AP&C according to their proprietary Advanced Plasma Atomization process (APA^{TM}). This process allows the creation of highly spherical powders with low internal porosity and almost satellite-free (Capus, 2017). In this work, the virgin powder directly supplied by the manufacturer was compared with the ones recycled more than 100 times, taken from the EBM machine after the sieving procedure. A specific number of recycles cannot be given since, in the EBM machine, the powder circulates permanently. The chemical composition of virgin and recycled powders is given in Table 1.

Table 1 - Chemical composition of the Ti-6Al-4V powders particles

	% C	% Fe	% V	% N	% Al	% O	% Ti
Virgin	0.01	0.205	4.03	0.02	6.50	0.11	Remaining
Recycled	0.01	0.202	4.03	0.03	6.46	0.30	Remaining

As can be seen, the oxygen percentage in reused powders is increased up to 0.3% and as a matter of fact, it has exceeded the value of 0.2%, which is defined as the limit value according to ASTM F2924 (Ghods et al., 2020).
The morphology of both powders was investigated using the Philips 505 Scanning Electron Microscope (SEM) in order to compare the external differences and understand the changes in terms of shape, roughness, and surface imperfections after recycling.
Besides the morphological characterization, the internal defects investigation was also carried out by evaluating the transverse cross section of the particles. For this characterization, a few powder particles of both batches were embedded in phenol-formaldehyde resin, and then the powder/resin samples were polished using a 0.3 µm Alumina suspension on porous woven wool felt, employing the standard polishing procedures. After the polishing process, both powder/resin samples were observed using an Optical Microscope.
As for post-processing investigations, in order to provide an average value regarding the porosities rates in the powder particles, a commercial software was used (NIH ImageJ, Bethesda, MD, USA). For each batch of powder, a total of 20 particles were analyzed starting from the optical images previously observed. Using the ImageJ software, the micro porosity were highlighted using a threshold command. Then the area of all the porosities was provided and it was compared to the total particle area using the relation (1).

$$\%\, defects = 100\ \frac{\sum A_{defects}}{A_{particle}} \tag{1}$$

3. Results and Discussions

3.1 Powder Morphology

Virgin and recycled powder morphology are shown in Figure 1. The virgin powder, being plasma atomized, have some few little imperfections, that are the tiny particles around the larger ones, which are known to lower the flowability of the powder (Seyda et al., 2012). The shape is almost spherical, with very few imperfections, and with a smooth surface, Figure 2. Some irregular particles may also be presented, although their presence is definitely low. The SEM image of the recycled powder is cleaner because almost all the satellites disappeared, which is beneficial to greater flowability. The disappearance of those tiny particles may be due to the temperature conditions, which is the preheating inside the EBM printing chamber that caused the melting of the satellites on the surface of the particles. The molten satellites contributed to increase the surface roughness of the reused particles, Figure 3. Some other defects due the temperature conditions are the elongated and irregular particles, while the broken ones are due to the mechanical conditions which is the sieving procedure.

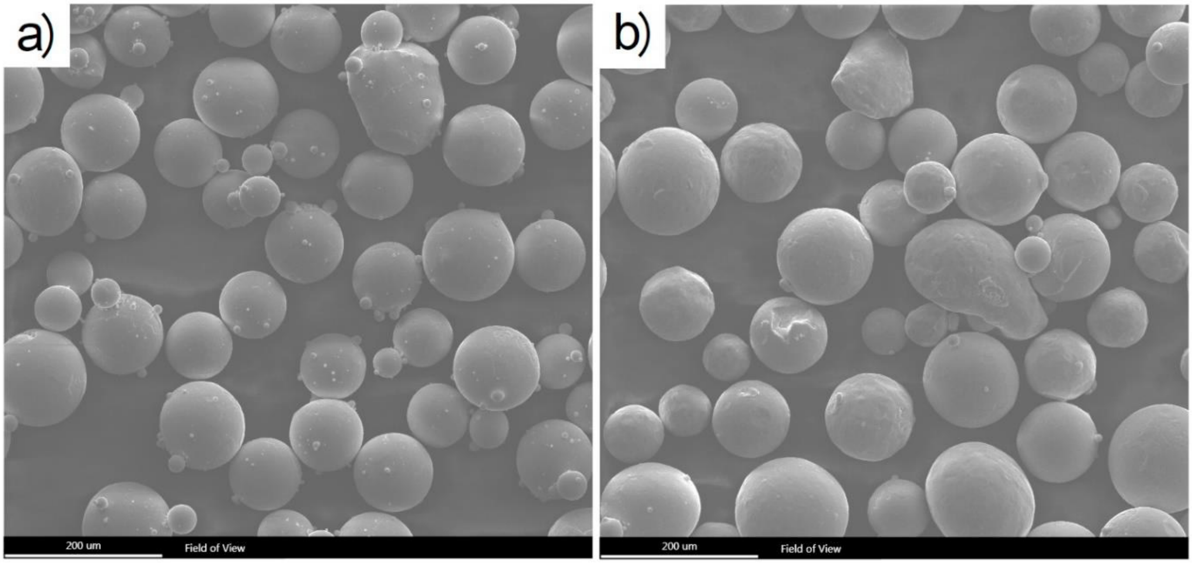

Figure 1: SEM observations of powder morphology: (a) virgin powder, particles have high sphericity with numerous tiny particles (satellites) around the larger particles, few irregular particles are also present. (b) recycled powder, particle surfaces are irregular and rough, almost all satellites disappeared, some particles are broken, elongated, or highly irregular in shape.

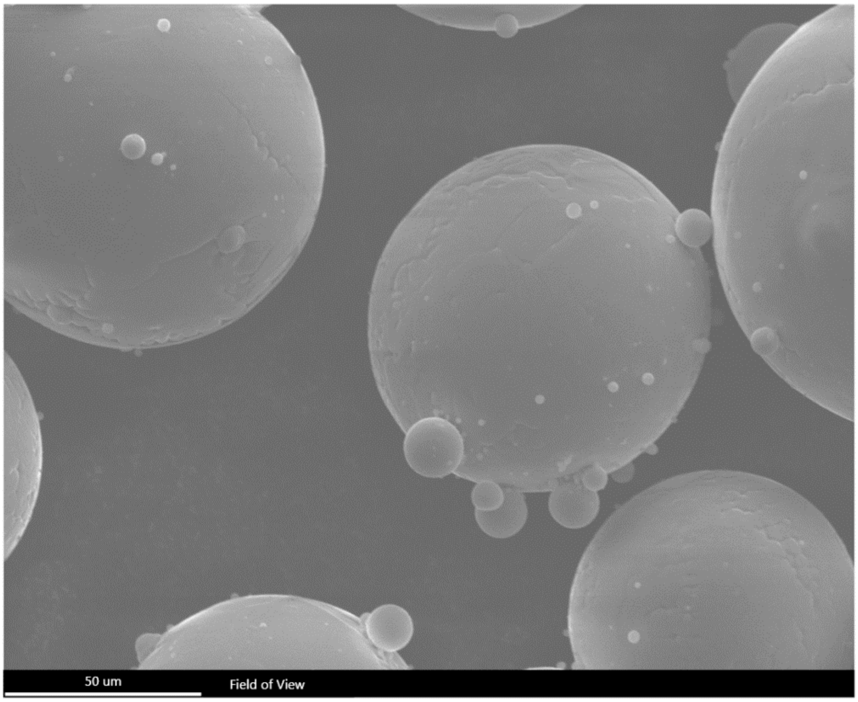

Figure 2: Surface characteristics of the virgin powder. The higher magnification allows observing the numerous satellites around the larger particles. The shape is almost spherical with few surface imperfections.

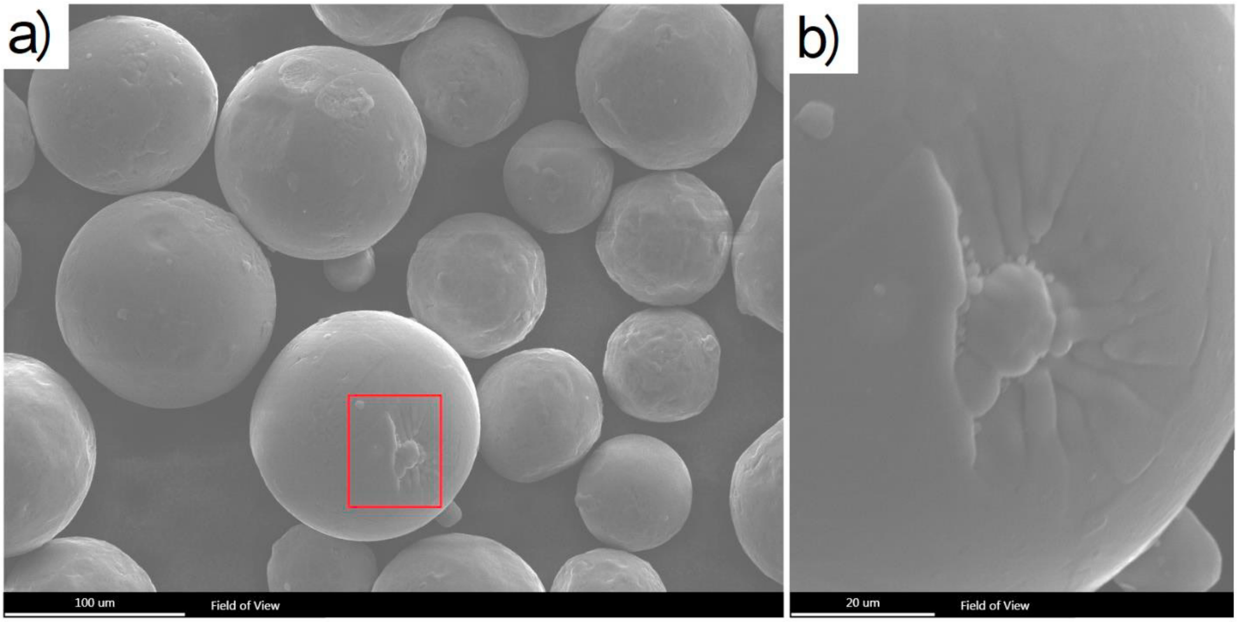

Figure 3: Surface characteristics of the recycled powder. (a) the higher magnification allows to better assess the surface roughness and the increased irregularity in shape. (b) surface imperfection.

3.2 Internal defects detection

Light optical photomicrographs of both batches of powders are shown in Figure 4. The light grey circular spots represent the cross-section of the powder particles, while the poorly visible darker spots (encircled in red) are particles that fell out during the polishing process.

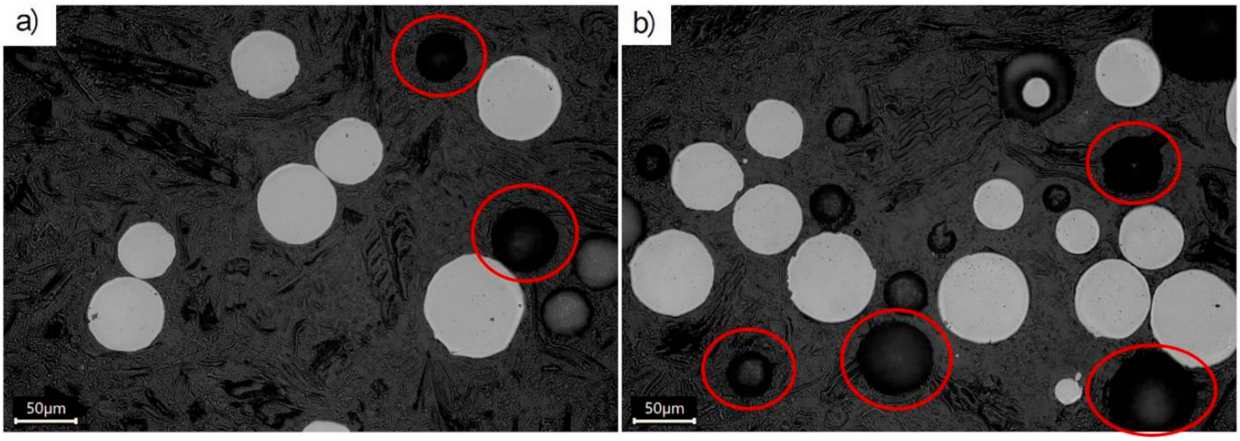

Figure 4: Optical images of powder particles' cross-section. (a) recycled powder. (b) virgin powder. In both batches, metallurgical pores and imperfections are present, with a higher number in the virgin powder.

The shape of particles is almost spherical for both batches, with some imperfect particles. The virgin powder contains more porosity which can be transferred to the final printed part, where even a small amount of porosity can be a problem for those applications where high strength is needed (Natali et al., 2019). The voids within the particles are

also spherical in shape, and this suggests that these pores contained gases from the atomization process, as it was also explained by some authors (Susan et al., 2006).
From this 2-D cross-section view, it is not possible to obtain information about the sizing of particles, since it is not possible to say if the diameter observed is the maximum one, and this is also true for the voids within particles, as it was well explained by some authors (Susan et al., 2006). This means that the areas of small pores are understimated.
Comparing the two powders with a high magnification FIG it is possible to clearly observe the disappearance of most of micro-porosities in the reused particles, due to the preheating and which made the cross-sectional view of the powders near fully dense.

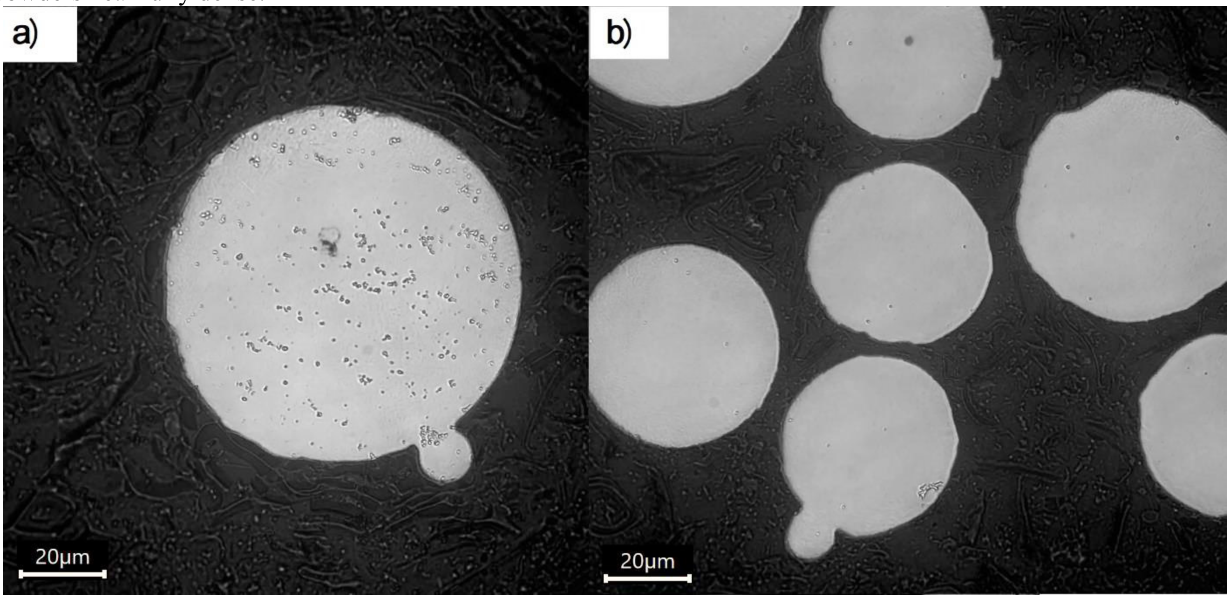

Figure 5 – Optical imageencircled in red of (a) virgin powder; (b) recycled powders.

3.3 Internal defects evaluations using ImageJ software

The percentage value of the internal micro porosities in the virgin powder was 3.030% as is shown in Figure 6. This value is consistent with the observed optical images, although these values are underestimated as from the cross-sectional view it is not possible to say if the diameter of the observed pores is the maximum one.

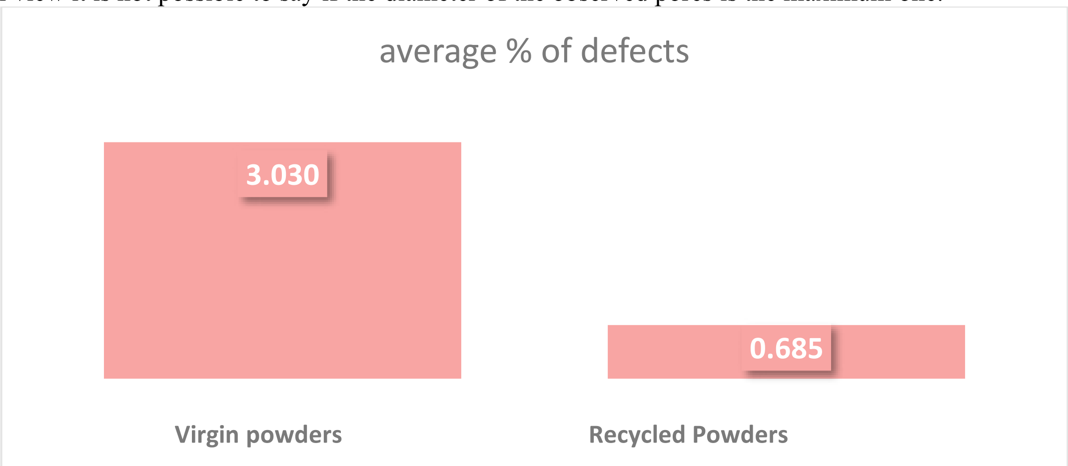

Figure 6:Average percentage of defects content in the virgin powder and in the recycled ones

4. Conclusions

The influence of recycling on powder morphology, external imperfections, and internal defects distribution was investigated by means of the Scanning Electron Microscope, the Optical Microscope, and the commercial software Imagej. The following results have been achieved:

- The virgin powder particles, being atomized using the Advanced plasma atomization process, already have some external and internal imperfections, such as micro porosities and satellites.
- From the SEM observations, after about 100 recycling cycles, almost all the satellites disappeared increasing the surface roughness but also increasing the flowability of the particles. Some broken and irregular particles also appeared.
- From the cross-section view, most of the internal micro-porosities disappeared in recycled powder, making the particles near fully dense compared to the virgin ones.
- ImageJ software was used to provide quantitative values which were consistent with the observed optical images. The provided values result to underestimate the actual situation since the diameter of the observed pores are not necessarily the maxima ones. For more detailed quantitative evaluations, the micro-computer tomography is more suitable.

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