Latest developments in Selective Laser Melting production of gold jewellery

Abstract

Selective laser melting (SLM) of layered gold powder is a rapid manufacturing (RM) technology capable of turning a CAD model into a jewel without intermediate steps. As with any new technology, giving concrete and tangible form to concepts requires research and development and as Strauss (SFS 2009) points out, issues that can arise with RM jewelry regard powder quality and availability.
This paper focuses on these issues together with laser power, laser scanning speed and the thickness of powder layer. A design of experiments (DOE) approach was used to characterize the influence of those variables on a 18K gold powder with reference to physical and chemical attributes, surface roughness, defects, and mechanical properties. A comparison of a series of specimens produced by SLM and lost wax casting will be presented.

Introduction

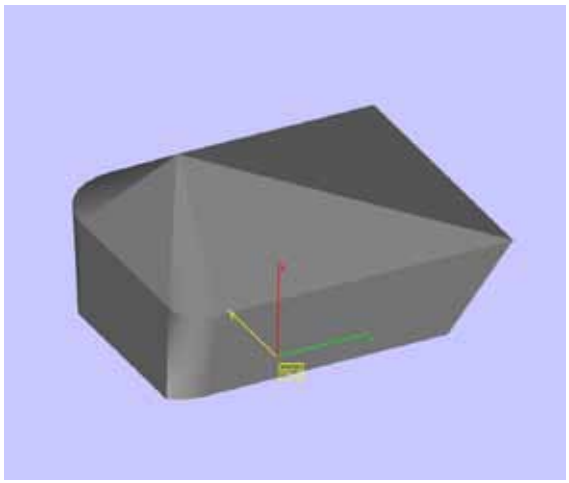
Rapid prototyping has been on the market for years and continues expanding. It has been rapidly adopted by several industrial sectors including jewellery thanks to the possibility to adapt to small batches. It's an innovative technology which allows complex geometrical items from a 3D CAD model prototype.
This technique is based on the idea that any object can be sliced into a series of layers of infinitesimal thickness therefore an object can be built in a consequential matter, layer after layer.
Since items are realized by continuously adding layers of powder, RP technology is also known as layer manufacturing technique.
Starting from powder alloys and using a laser which follows a defined 3D drawing, items with shape and thickness too difficult to realize with other techniques can be achieved.[1]
Amongst techniques whose starting point is the drawing of a 3D object, SLM[2], which stands for Selective Laser Melting, is clearly the technique which lends itself more for the production of items where surface esthetical qualities and high physical-mechanical characteristics are required. The resulting structure is similar to that of a typical investment casting product but with an extremely small grain size due to the marked undercooling of the alloy.
This paper covers three main areas, i.e. surface roughness investigation, defects analysis, physical-mechanical properties.
The variables are laser power, laser scanning speed, powder layer thickness and powder particle-size distribution.

More specifically, an 18ct yellow gold powder in 3 different particle-size ranges was used in this study. Specimens were realized with this powder and tests were

consequently carried out.
Our purpose was to highlight, using a DOE (Design Of Experiments)[3, 4] approach, not only the effect of the single variables which play a crucial role in conferring certain characteristics to the product , but also the interaction among them. This method is useful to constantly improve product quality and process efficiency.

Experimental section

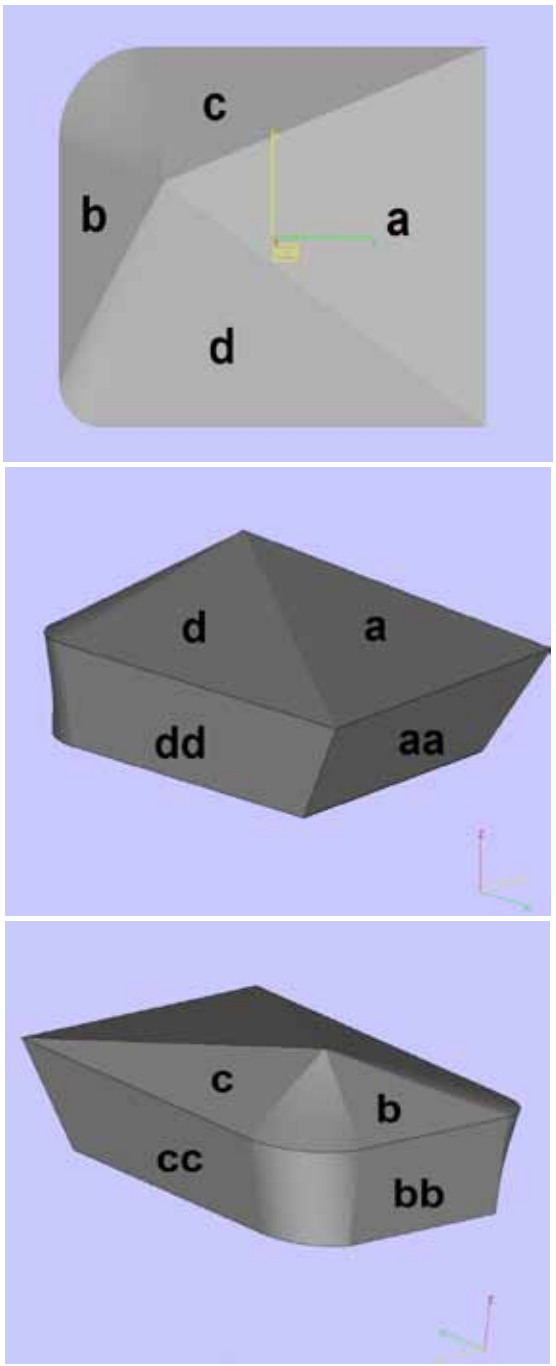
Studies were conducted using a Realizer - SLM 50 machine with 70mm diameter working platform and maximum working height of 40mm. The machine features a 100W fibre laser with 10 µm spot size operating in an Argon protected atmosphere.
The powder used in the tests was produced in our laboratories using a gas atomizer which works in a controlled atmosphere (the cycle features complete vacuum of atomization and melting chambers and then restoring of 1 bar pressure using Argon gas). This type of atomization allows particles with narrow distribution, spherical morphology and completely dry powders, necessary conditions for metal powders to be used with the SLM process. In this respect, it is known that the aforementioned characteristics influence powder flowability significantly, an essential feature for good quality powder layer which, during processing, must be laid down on the working platform.
An alloy with the following composition was therefore chosen for this study: 75% Gold, 11.75% Silver, 0.5% Zinc, 0.01% Iridium. It complies to 3N colour as defined by ISO EN 28654 norm.
The surface study of raw items followed a study on the type of specimen to use. The definition of a structure which could enable analysis of roughness variation on specimens realized with SLM was not easy. The key point of our study was to design a specimen with surfaces with different inclinations to measure differences among those very same surfaces, in addition to realizing a small volume specimen to reduce powder necessary for its realization.
Al specimens produced were designed using Rhinoceros software and supported using Magics software from Materialise.
The specimens on which surface roughness trials and metallographic analysis were conducted (see picture 1) was 0.30 mm thick, 9.21 mm high, had a volume of 200.45 mm^3 and surfaces with different inclination.



Picture 1 – 3D drawing of specimen, displayed using Magics software.

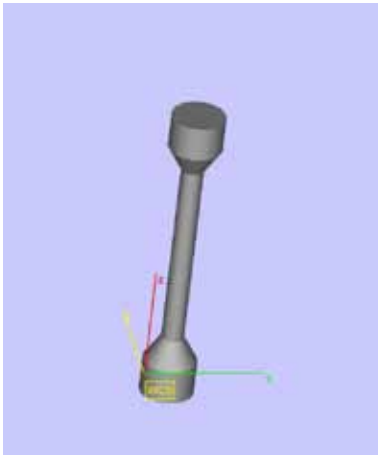
The specimen was designed to allow surfaces to have a particular inclination compared to the working platform to allow studying of surface roughness variation depending on inclination.
Surface nomenclature is in picture 2. The value of angles subtended between surface

and working platform is as follows: a=14°; b=37°; c=33°; d=20°; aa=60°; bb=90°; cc=76°; dd=76°.



Picture 2 – Specimen surfaces displayed using Magics software.

Five test pieces for tensile strenght test were produced together with the specimen subjected to roughness analysis to verify the quality of the structure built.
Tensile strenght specimens have the following dimensions: 25.90 mm height, 15.77 mm working length, 157.73 mm^3 volume. This specific geometry was chosen after a series of tests on specimens with different shape. The tensile strenght test piece used is shown in picture 3.

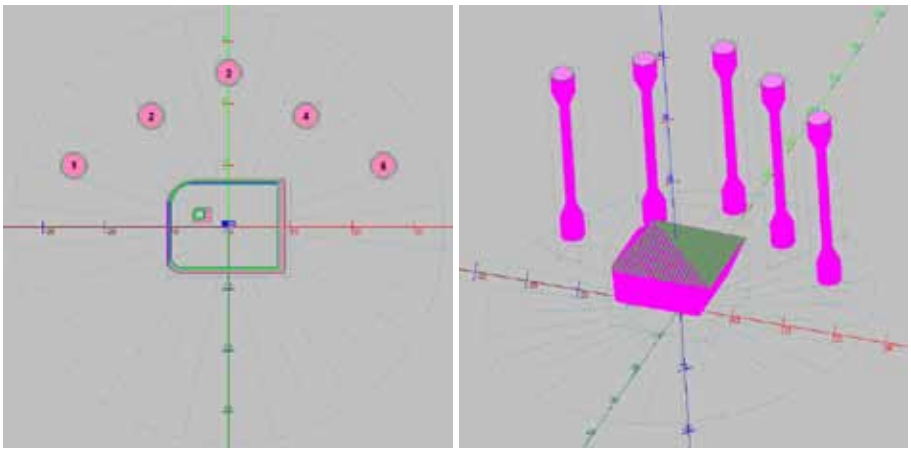


Picture 3 – 3D drawing of tensile test piece, displayed using Magics software.

All specimens realized were placed in designated places on the working platform, i.e. specimen in picture 1 at coordinates x=0 mm and y=0 mm, whereas tensile test specimens were placed at coordinates specified in table 1.

Table 1 – Coordinates of tensile test specimen on working platform

Tensile test specimen	X (mm)	Y (mm)
1	-25	10
2	-12.5	18
3	0	25
4	12.5	18
5	25	10

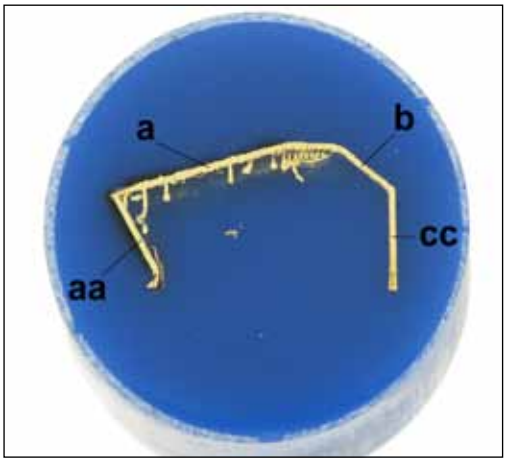


Picture 4 – 2D and 3D representation of specimens realized on working platform displayed using Realizer software.

Sixteen trials were scheduled, each associated with a specific particle-size distribution of powder used, layer thickness, laser power and scanning speed. When the last three factors are known, the specific energy associated can be determined. More specifically, powder in three different particle-size distributions (named A, B and C) was used together with two different layer thickness (40 and 50 µm), different laser power (80 and 90 W) and scanning speed (0.25 and 0.30 m/s). All low values are indicated as (-1) and high values as (+1).
Table 2 shows the program used in this study. B particle-size distribution is the merging of two intervals, A and C. Layer thickness was changed on two layers (-1 the lower layer, +1 the upper layer) only for particle-size distribution A. Laser power and scanning speed were changed on two levels.

Table 2 – Work program					
Particle-size distribution	Layer thickness	Power	Scanning speed	Specific energy (106 J/m^2)	Test denomination
A	–1	–1	–1	8	A-1
			+1	6.7	A2
		+1	–1	9	A3
			+1	7.5	A4
	+1	–1	–1	6.4	A5
			+1	5.3	A6
		+1	–1	7.2	A7
			+1	6	A8
B	+1	–1	–1	6.4	B1
			+1	5.3	B2
		+1	–1	7.2	B3
			+1	6	B4
C	+1	–1	–1	6.4	C1
			+1	5.3	C2
		+1	–1	7.2	C3
			+1	6	C4

Surface study of items after construction was conducted using Taylor Hobson FORM TALYSURF INTRA2 surface roghness tester.
For each surface, picture 2, 2D and 3D analysis were carried out. The latter were possible thanks to a working platform which enabled 16 mm^2 surface mapping for “a”, “d”, “aa”, “bb”, “cc”, “dd” surfaces and 4mm^2 for “b” and “c” surfaces.
Tensile test specimens were subject to density analysis and tensile tests to better evaluate their physical and mechanical properties.
Density trials were conducted using Sartorius scales which features a device to determine density, whereas tensile tests were carried out using INSTRON universal dynamometer with 2kN load cell.
After analysing the surface, samples were cross sectioned - as can be seen in picture 5 – and prepared for metallographic analysis. Sections “a”, “b”, “bb”, “aa” were studied at the metallographic microscope to verify the microstructure of 16 trials.



Picture 5 – B3 Sample specimen after metallographic etching

To compare SLM technology with current techniques used in jewellery production we realised the same samples using traditional and direct investment casting technique. Each of the two cast trees included six tensile test specimens and three specimens which had to undergo surface roughness testing (the latter with 0.5 mm thickness each instead of 0.3mm as with specimens realized with SLM). The procedure followed is:

1. Pre-melting with induction melting furnace. Metal was mould cast at a temperature of 150°C above liquidus temperature. Then the ingot was rolled and cut to facilitate the next processing stage.

2. Casting was carried out in a vacuum induction casting machine. Casting temperature was 150°C above liquidus temperature. Flask temperature was 700°C. Flask were rapidly quenched in water after 300 seconds.

As with tests carried out on specimens realized with SLM, investment casting specimens underwent roughness analysis, tensile tests, metallographic analysis.
Based on the results from the 16 trials a final test with SLM, using B particle-size distribution powder, different layer thickness, laser power and scanning speed - duly optimized to achieve the best results possible in terms of roughness and density values - was planned.
The results were then compared with specimens produced using investment casting technique.

Results and discussion

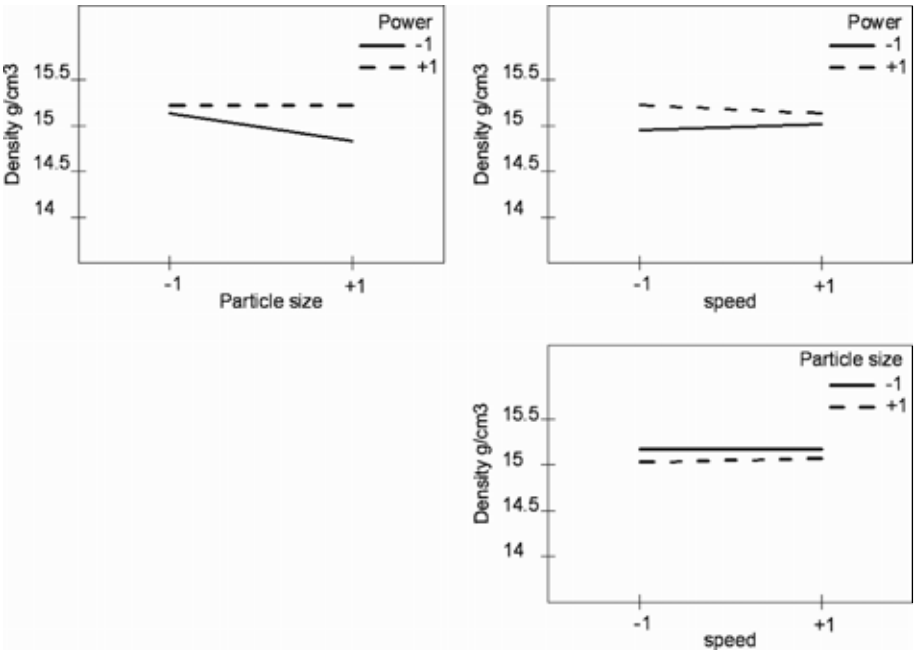
Density

Density of tensile specimens is in Table 3.

Table 3 – Density of tensile specimens

Test denomination	Density (g/cm^3)	Test denomination	Density (g/cm^3)
A1	15.19	B1	15.14
A2	15.18	B2	15.20
A3	15.23	B3	15.21
A4	15.22	B4	15.21
A5	15.11	C1	14.79
A6	15.14	C2	14.94
A7	15.20	C3	15.23
A8	15.19	C4	15.14

All A and B specimens have a density between 15.11 and 15.23 g/cm^3, which is very close to the the alloy bulk density. Differences within these two particle-size distributions are very modest. It can be inferred that for A and B particle-size distributions no effects of process parameters on density can be observed.
In the case of C specimens, the two specimens produced with lower laser power (C1 and C2) have lower density which is representative of residual porosity. The results of statistical analysis are reported in picture 8 to highlight the effect of particle-size analysis comparing specimens A (Level -1) and C (level +1), thickness being the same. Ordinate scale is magnified to show small but significant differences noted. Please note the interaction between granulometry and laser power when determining density.



Picture 8 – Process parameters effect on density of specimens produced with A and C particle-size distribution.

Tensile test results

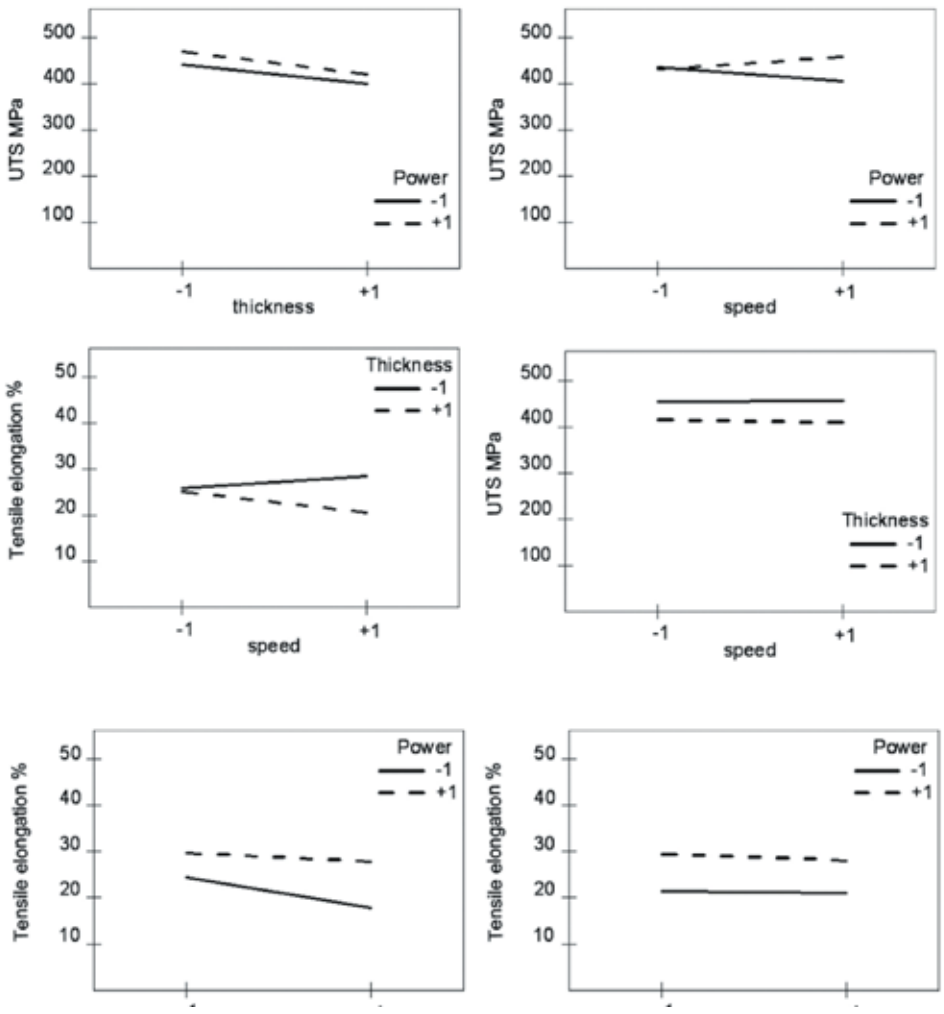
Tensile tests were carried out to evaluate material soundness. In case of ductile materials, Ultimate Tensile Strength (UTS) and elongation (Ɛ), which is significant with ductility, are the most relevant aspects. Their values are reported in Table 4.

Table 4 – Tensile test results

Test denomination	UTS (MPa)	Ɛ (%)	Test denomination	UTS (MPa)	Ɛ (%)
A1	444.0	23.3	B1	435.6	20.8
A2	438.0	25.7	B2	404.9	20.6
A3	464.0	28.2	B3	402.4	28.5
A4	474.0	31.1	B4	457.6	23.7
A5	427.0	19.5	C1	347.8	12.9
A6	372.0	16.2	C2	333.6	14.7
A7	399.0	30.6	C3	390.4	29.6
A8	442.0	25.0	C4	393.3	16.5

From a general point of view it can be inferred that specimens A have basically superior properties compared to specimens B only if produced with thickness on lower level, whereas specimens C produced with lower laser power (C1 and C2) are the worst ones. This observation seems to be corroborated by density, as mentioned before.

In other cases, instead, densities are very similar thus making it interesting to evaluate the effect of experimented process parameters. Results of statistical elaboration are shown in pictures 9 and 10. Picture 9 refers to specimens A to highlight the effect of layer thickness, laser power and scanning speed on resistance and ductility achieved using particle-size distribution A.



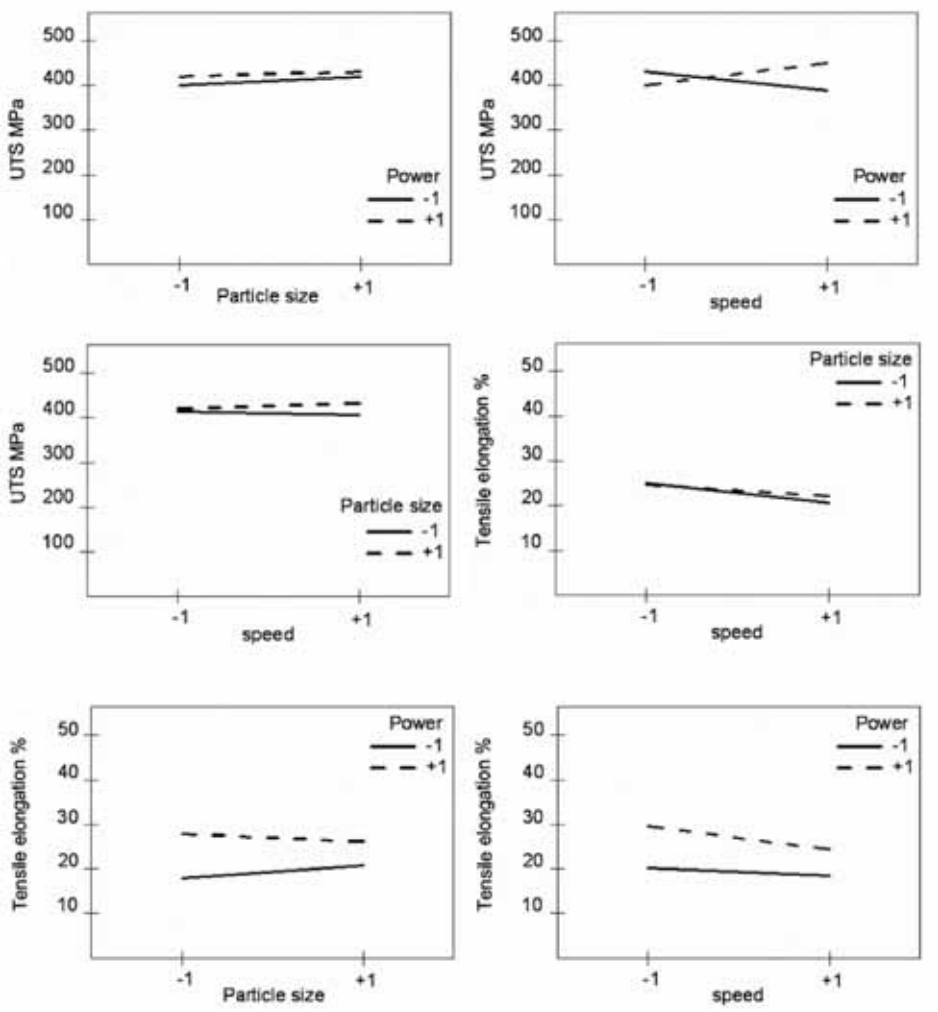
Picture 9 – Effect of process parameters on tensile strength and elongation of specimens produced with particle-size distribution A.

It must be noted that:

a) By increasing power tensile strength increases little but ductility more significantly;

b) Layer thickness influences tensile strenght (by increasing thickness, resistance decreases) and ductility only when speed is high (by increasing thickness, ductility decreases).

Picture 10 shows data elaborated using thickness of upper layer to highlight the effect of laser power, scanning speed and particle-size distribution comparing intervals A (-1) and B (+1).

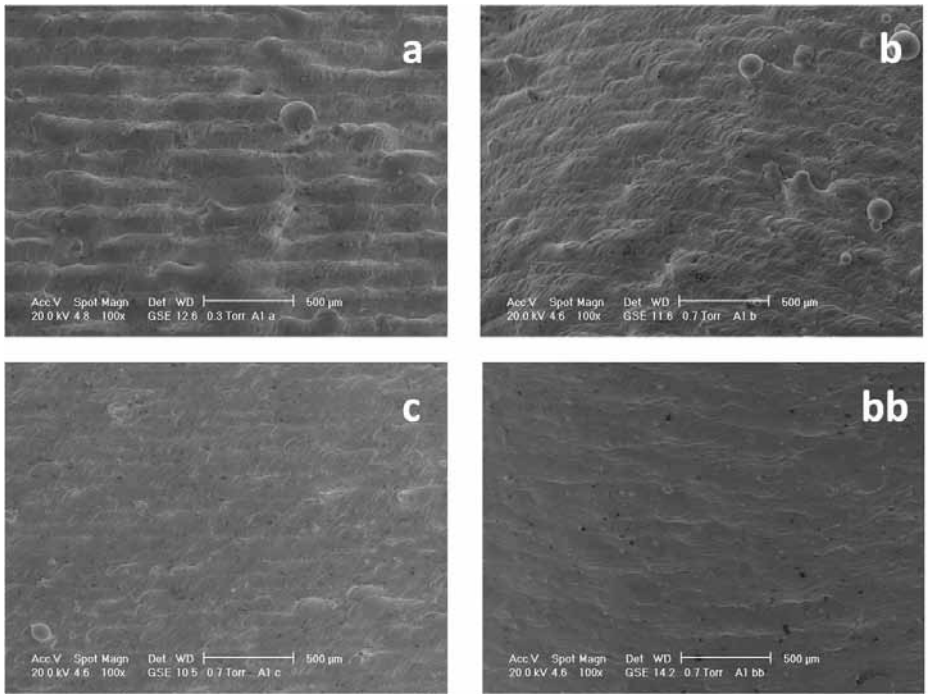


Picture 10 – Effect of process parameters on tensile strength and elongation of specimens produced with A and B particle-size distributions.

It must be noted that granulometry does not influence mechanical characteristics; laser power and scanning speed effect confirms the previous results.
Statistical elaboration with C specimens is pointless: the density of two specimens is clearly lower than that of the rest, thus ruling over the possible effect of process parameters.

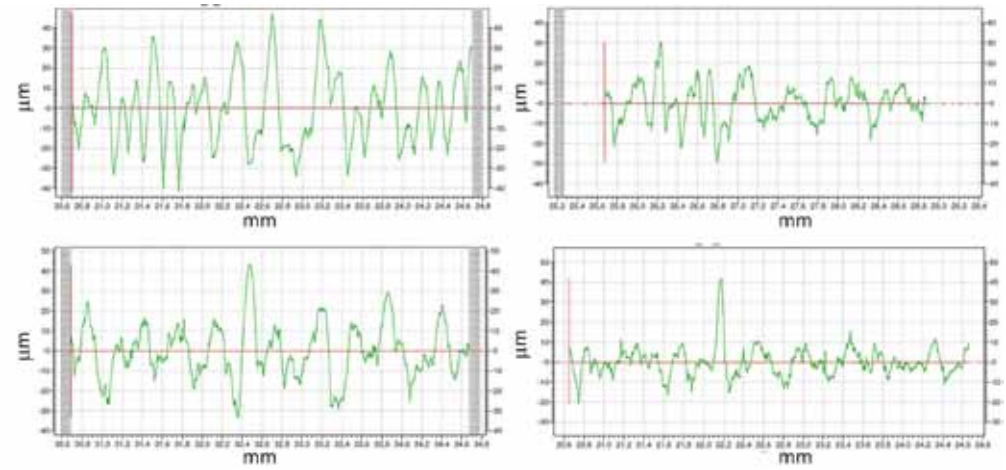
Surface morphology

The morphology of the surface of specimens depends on their inclination to the table. Picture 11 shows, for examples, surfaces “a”, “b”, “c” and “bb” of the specimen A1. A wavy morphology can be observed on all surfaces: it can be traced back to the molten pool solidified during piece manufacturing. The different height of the surface relief is clearly visible. Non-melted particles of powder which remained bound to the surface due to solid-phase sintering can be observed.



Picture 11 – Morphology of "a", "b", "c" and "bb" surfaces of specimen A1.

An example of surface profile recorded on the four surfaces of picture 11 is shown in picture 12. Roughness were processed to achieve the distinctive features of surface profile: Ra, Rv, Rp, and Rt. These parameters have a different meaning and are preferably used based on application of items produced. E.g. in the case of surfaces subject to wear and tear, Ra is universally used to characterize roughness; with lubricated wear and tear, Rv is very significant as it represents how deep the valleys – which act as lubricant tanks - of the profile are; Rp represents the height of emerging peaks compared to the average line of the profile and the material removed during run-in stages. In the case above it is more appropriate to refer to Rt which, by representing the total height of the profile, represents the thickness to be removed to achieve a perfectly smooth surface which is aesthetically acceptable.

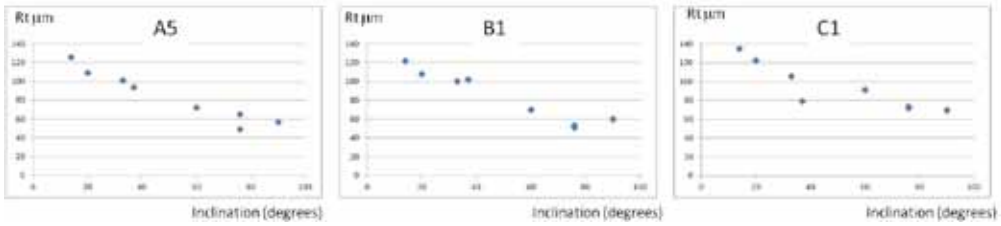


Picture 12 – Profile of surfaces "a", "b", "c" and "bb" of specimen A1.

Picture 13 shows the influence of inclination as to working platform: in this case Rt is in function of the inclination angle for specimens A5, B1 and C1 which were produced with the same process parameters, but different particle-size distribution of the powder. It must be noted that:

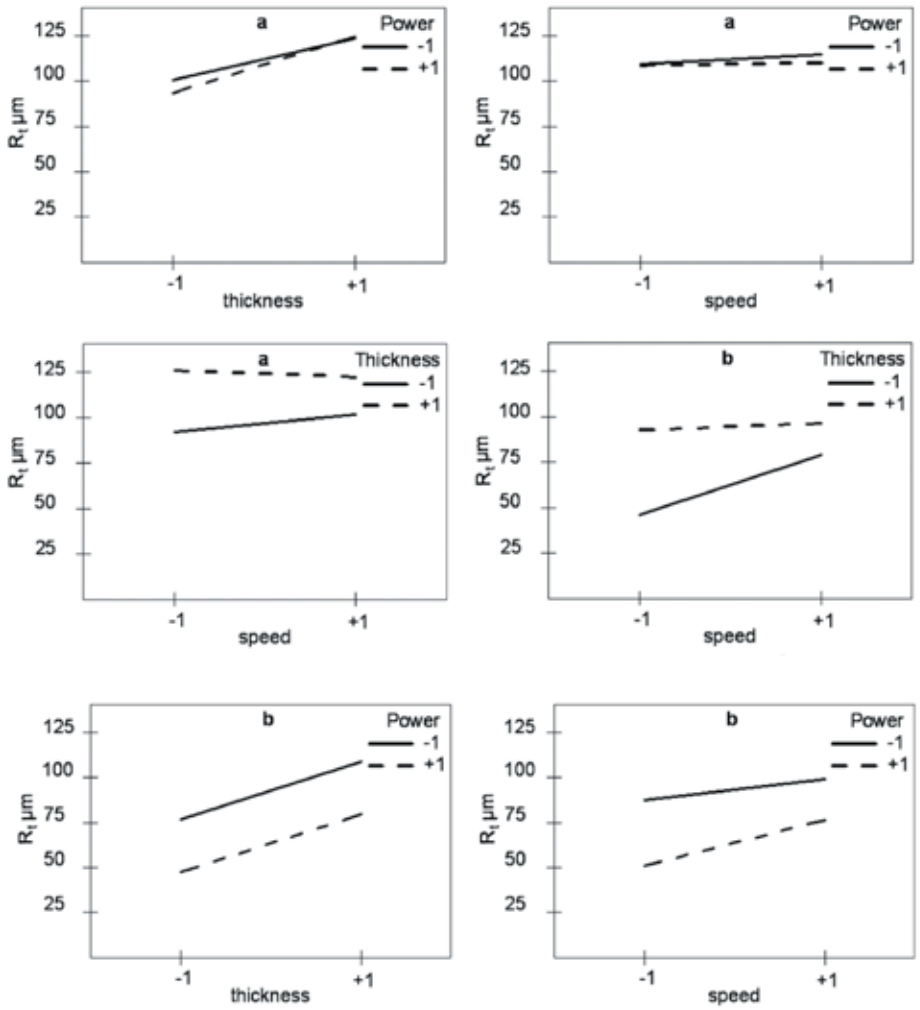
a) There is a direct correlation between inclination and roughness value

(higher Rt values measured on less inclined surfaces);
b) Increased differences were noted among less inclined surfaces;
c) Layer thickness being equal, Rt values are similar for all particle-size distributions.

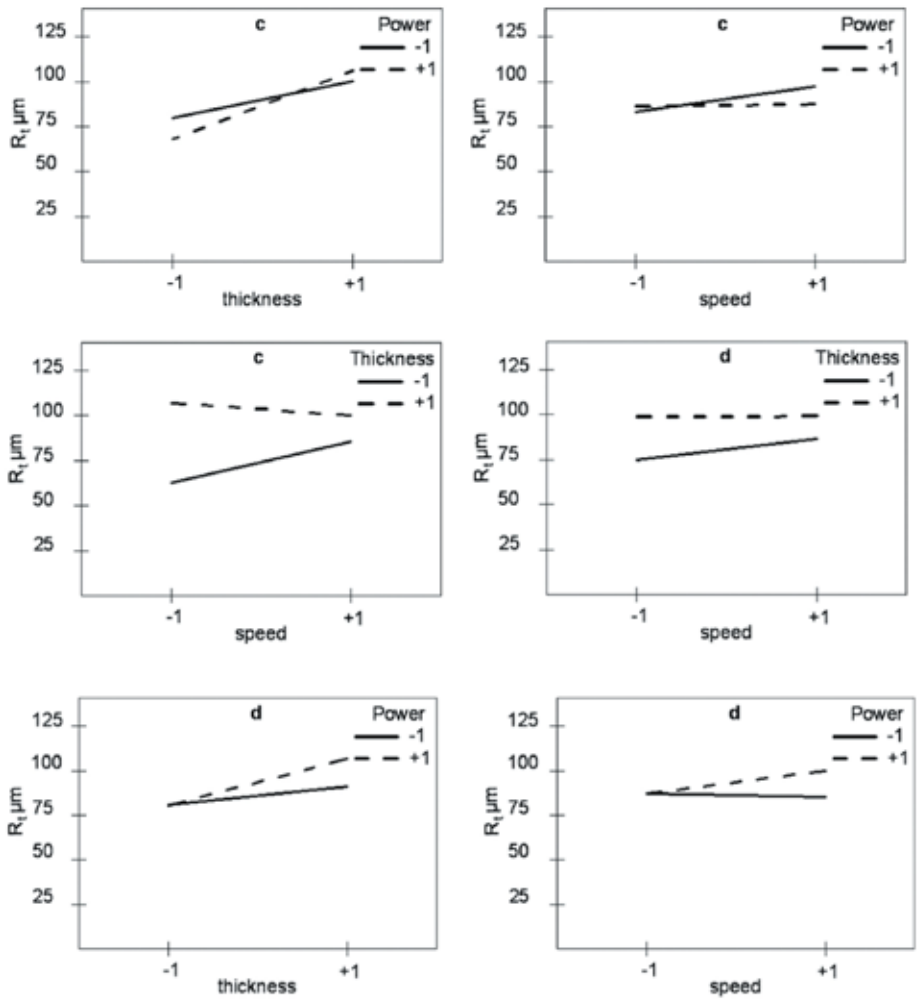


Picture 13 - Influence of surface inclination on total Rt roughness.

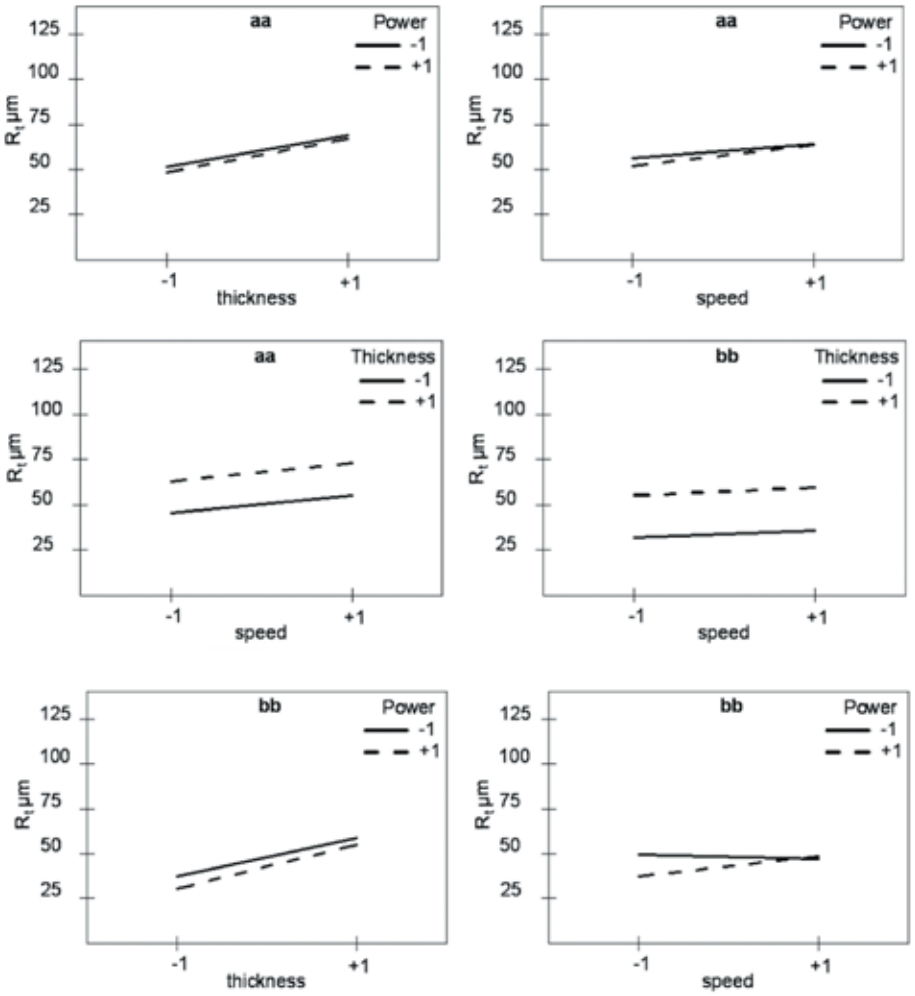
Rt values were statistically elaborated to highlight the effect of process parameters. Elaboration was carried out for all eight surfaces measured and the results are as follows.
Pictures 14-17 report the result of elaboration for specimens produced with particle-size distribution A to underline the effect of laser power, layer thickness and scanning speed.



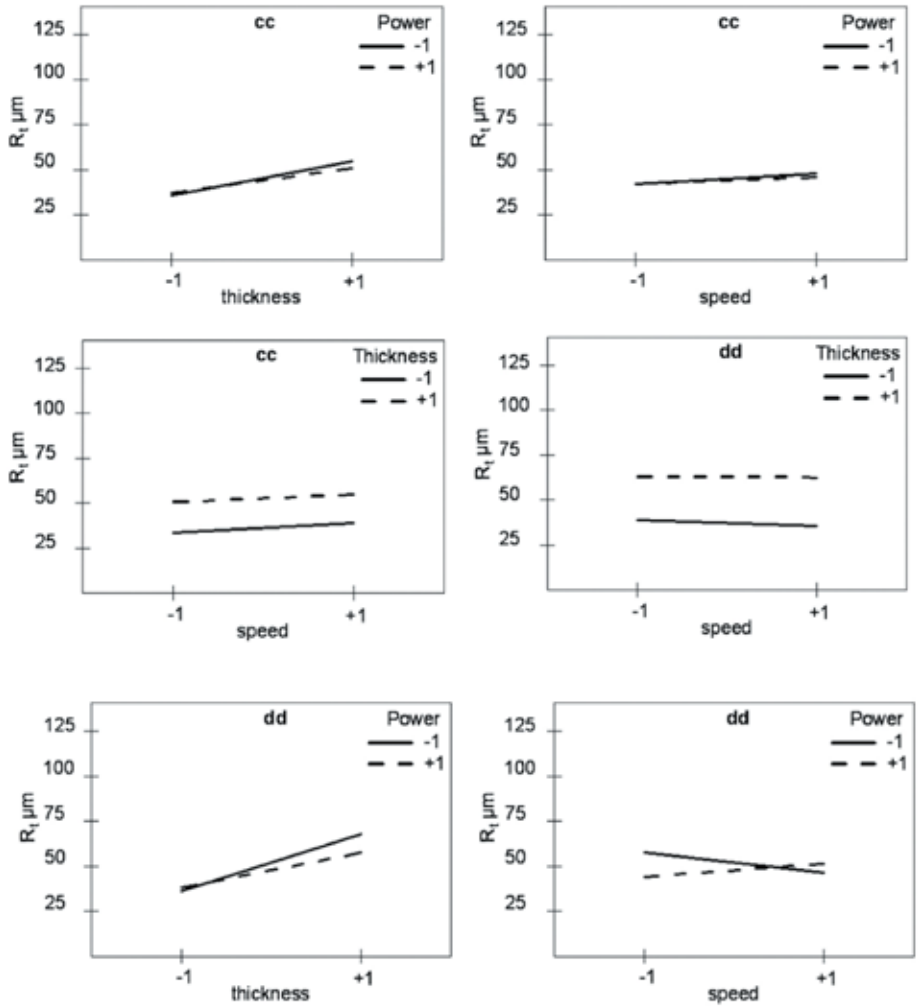
Picture 14 – Effect of process parameters on total Rt roughness of surfaces "a" and "b" of specimens produced with particle-size distribution A.



Picture 15 – Effect of process parameters on total Rt roughness of surfaces “c” and “d” of specimens produced with particle-size distribution A.

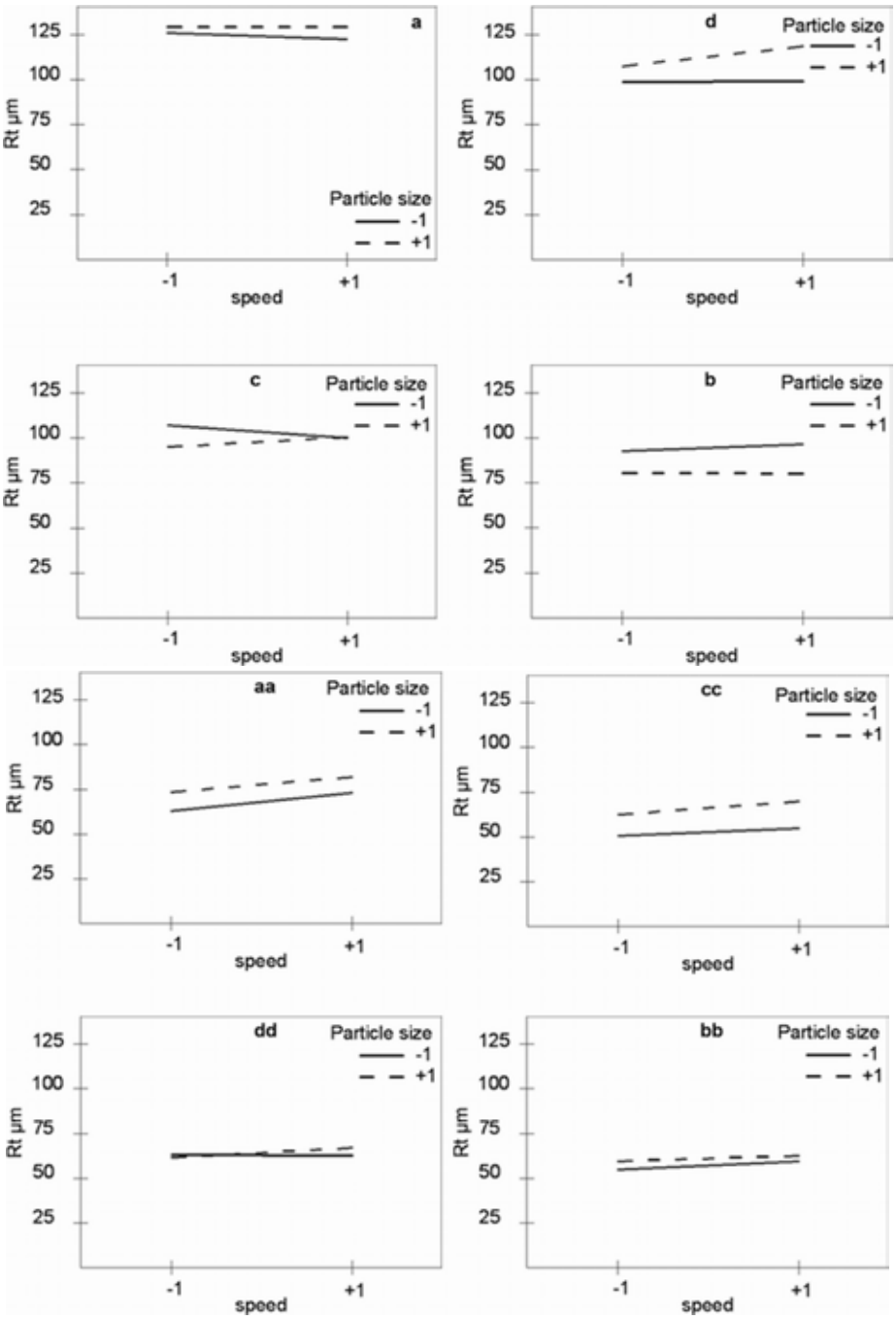


Picture 16 – Effect of process parameters on total Rt roughness of surfaces “aa” and “bb” of specimens produced with particle-size distribution A.



Picture 17 – Effect of process parameters on total Rt roughness of surfaces "cc" and "dd" of specimens produced with particle-size distribution A.

Following careful examination of the pictures, the following results can be observed: Rt increases when thickness on all surfaces and power on "b" surface increase.
Picture 18 shows the results of statistical elaboration on A and C specimens to highlight the effect of particle-size distribution, laser power and scanning speed (in this case layer thickness is constant on the upper level).



Picture 18 – Result of statistical analysis on total roughness of specimens A and C.

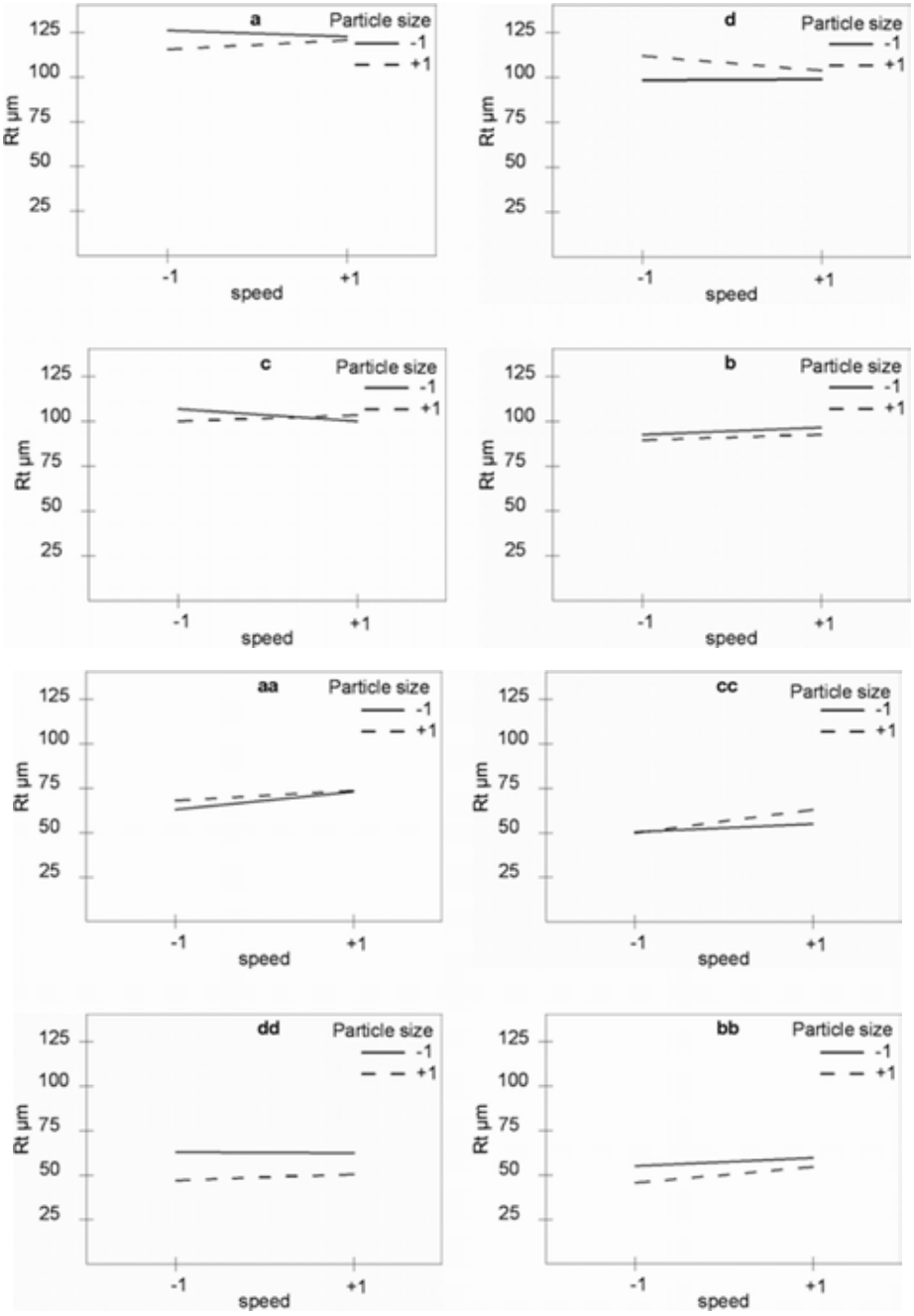
It can be observed that:

a) Rt values show the effect of granulometry: they are generally lower for specimens A, except when surfaces have an inclination of 33° and 37° ("b" and "c");

b) The effect of granulometry is less marked for surfaces only slightly inclined ("a") and very inclined ("dd" and "bb");

c) The influence of speed and power is negligible. Influence of power on "b" and "bb" surfaces can be observed.

Picture 19 shows the results of statistical elaboration of specimens A and B to highlight the effect of particle-size distribution, laser power and scanning speed (layer thickness is constant on the upper level in this case, too).



Picture 19 – Result of statistical analysis of total roughness of specimens A and B

It can be observed that:

a) Rt roughness values are similar for A and B and appear to be little influenced by granulometry;

b) Effect of granulometry can be seen only on surfaces inclined the most (“dd”, “bb”);

c) On such surfaces roughness values for B specimens are lower;

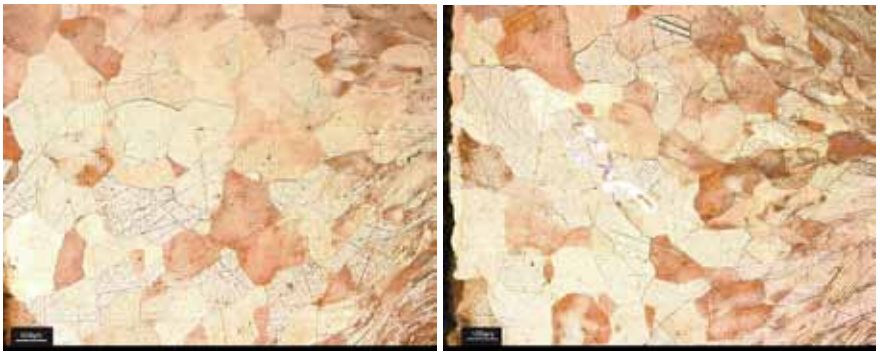
d) The influence of speed and power is negligible. Effect of power on “b” surface can be observed and can be traced back to the one observed during analysis of individual A specimens.

Comparison between SLM and investment casting

To better understand the difference in terms of microstructure between items realised with SLM and items realised with technologies currently used for the production of jewellery, specimen B3 micrography as an example of surface observed following SLM process is shown below.

“bb” section

Difference of grain structure among 16 tests is not evident. It must be noted that in all sections of specimens observed with SLM technology grain size is small, the clear result of using a 10 μm spot laser which melts a layer of powder with infinitesimal thickness. Melting therefore involved a very small volume of material.
Pictures of investment casting specimens taken with metallographic microscope are shown below.



Traditional Casting “bb” cross-section **Direct Casting “bb” cross-section**

Picture 21 – Cross sections after metallographic etching.

The difference of grain structure of specimens realized with SLM technology and specimens realized with investment casting is evident. Grain size in SLM specimens is around 10 microns, whereas grain size of investment casting specimens is around 90 microns. Columnar grain structure was observed for SLM specimens.
Table 5 shows data of SLM specimens realized in the final test - its parameters are the result of the statistical analysis carried out – and specimens realized with traditional and direct casting – in terms of surface roughness.

Table 5 – Surface roughness

	Traditional investment casting	**Direct investment casting**	**SLM Final test**
Surface	**Rt**	**Rt**	**Rt**
a	14.6	35.7	59.1
b	13.0	20.4	22.1
c	10.8	23.0	25.2
d	15.9	20.4	33.7
aa	23.5	23.5	27.5
bb	26.6	18.6	26.5
cc	39.9	31.8	27.1
dd	31.9	44.9	29.1

A comparison of roughness values reveals that with SLM technology Rt values are close to those of direct casting specimens. For some surfaces, i.e. “aa” “bb” “cc” and “dd”, surface roughness can be compared to specimens realized with traditional investment casting.
Eventually, Table 6 shows data concerning comparison between SLM specimen realized in the final test and specimens realized with investment casting in terms of density, tensile strength and elongation.

Table 6 – Density and tensile test results

	Traditional investment casting	**Direct investment casting**	**SLM Final test**
Density (g/cm³)	15.26	15.22	15.24
UTS (MPa)	414.9	395.9	474.2
ε (%)	42.4	38.5	33.5

All specimens feature density between 15.22 and 15.26 g/cm^3, which is very close to the bulk density of the alloy. Therefore it can be deduced that residual porosity is very limited. The data collected from the analysis clearly shows that specimens realized with SLM technology have higher tensile strength but lower ductility compared to specimens realized with investment casting.

Conclusions

This study clearly shows how feasible the production of gold jewellery with SLM technology is. Choice of optimal process parameters and particle-size distribution enables products with a density very close to the real one which feature no porosity – even micrometrical. The result of this can be seen not only on material soundness – as shows by tensile tests – but also by the possibility of achieving surfaces which are aesthetically excellent once the rough surface layer is removed. It is truly impossible to achieve a smooth surface with SLM due to the particular way items are produced.
Surface morphology depends on the inclination of surfaces to working platforms and on process parameters. Thanks to a DOE approach it was not only possible to identify the effect of process parameters but also to select those which enable lower surface roughness.
A relevant result was reported concerning the effect of granulometry of the powder used. Specific energy being equal, granulometry influences the amount of material influenced by the thermal contribution and consequently molten pool shape, with important effects on the morphology of the surface.
The comparison between SLM technology and investment casting highlights smaller grain size in specimens realized with SLM. This could explain the higher tensile strenght detected in the final test. Density values and porosity values in the same range on surfaces below a certain inclination degree also emerge.
Therefore this study only represents the starting point in the study and in-depth analysis of SLM technology applied to jewellery. Further research will be carried out in the future on new variables such as chemical composition of powders.

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