Author's Post-print version
Article reference: http://dx.doi.org/10.1016/j.cirp.2014.03.060

Contents lists available at SciVerse ScienceDirect

CIRP Annals Manufacturing Technology

Journal homepage: www.elsevier.com/locate/cirp

High cycle fatigue properties of selective laser sintered parts in polyamide 12

Brecht Van Hooreweder, Jean-Pierre Kruth (1)

KU Leuven, Mechanical Engineering Department, Belgium

Selective laser sintered parts in polyamide are increasingly being used in functional applications. The monotonic properties of these parts are well-known and documented. The cyclic material properties, on the other hand, are largely unknown. Therefore, in this paper a systematic analysis is made of fatigue properties and failure mechanisms of selective laser sintered parts subjected to fluctuating loading in tension/compression. Both plain and notched specimens are produced along perpendicular building directions. The fatigue behaviour of these parts is analysed and compared to injection moulded samples. In addition, the influence of the test frequency and the notch manufacturing method is studied.

Additive Manufacturing, Selective Laser Sintering, Fatigue

1. Introduction

During recent years, Selective Laser Sintering (SLS) has been frequently used to produce functional parts in polyamide 12 (PA12). Compared to conventional production techniques for polymers such as injection moulding (IM), the SLS process offers advantages including a short design to manufacturing cycle time, high geometrical freedom, customised components and inexpensive production of small numbers of parts [1]. However, the long-term mechanical properties and corresponding failure mechanisms of SLS-PA12 parts are at present not fully understood. This is also illustrated in a recent review paper by Goodridge et al. [2].

In previous work by the present authors, the influence of a dynamic tension/compression load on SLS-PA12 parts was analysed and related to the density, crystallinity, and microstructure of the material [3,4]. It was found that the material density is a crucial factor influencing the fatigue life. The lower the density, the more unfused powder particles and the higher the chance of crack nucleation. In addition, the failure mechanism under repeated loading was found to be of thermo-mechanical nature and increased fatigue resistance of the notched specimens was noticed compared to the plain samples. In this paper, the previous work is extended and the influence of the test frequency and the manufacturing method of the notch is studied.

2. Methodology

Plain and notched cylindrical specimens with sample geometries shown in Figure 1 were produced with both SLS and IM as illustrated in Figure 2. The laser sintered parts were manufactured along two perpendicular building directions, SLSx and SLSz, using an optimal set of production parameters [5]. All SLS specimens were built at the same location in the build platform to limit variability in the properties of the parts. Also, one unique batch of PA12 (EOS PA2200) powder was used for both SLS and IM. Granules for injection moulding were made by cold isostatic pressing of PA12 powder in rods (Ø10mm) that were cut in thin slices (granules). Consequently, the thermal history, melting temperature (186°C), and crystallinity (45%) of both PA12-forms are the same [5], and a real comparison between SLS and IM parts can be made as shown in Figure 2. The average diameter of the PA12 granules and the PA12 powder was measured to be 10mm and 60µm respectively.

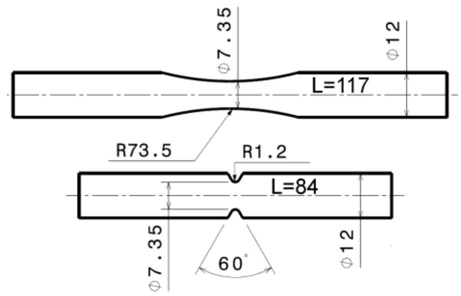

Figure 1. Plain and notched cylindrical specimen geometry.

Fatigue tests were performed using a servo-hydraulic test rig in force controlled tension/compression loading at 3Hz and 1Hz. No mean stress and no artificial cooling were applied. Surface temperatures were measured during fatigue testing using thermocouples and an IR-camera. The stress-based fatigue approach was used since the expected mechanical service load is low and fatigue life of PA12 parts is likely to be dominated by the nucleation of cracks and not by crack propagation.

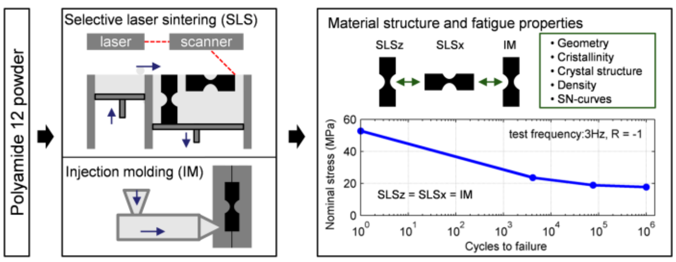

Figure 2. Comparison of the fatigue behaviour of SLS-PA12 and IM-PA12.

3. Results and discussion

3.1. Specimen properties

Some fatigue influencing factors of the IM parts and of the horizontally (x) and vertically (z) built SLS parts are shown in Table 1. The measured test diameters are taken into account to determine the exact stress in the smallest cross-section of the samples. Thanks to the optimal production parameters, the SLS parts are nearly full-dense. This leads to high, nearly isotropic, UTS strength values, which are similar to the UTS of the IM parts. All UTS values are slightly higher than values reported in literature: 48MPa for SLS-PA12 [2] and 45MPa for IM-PA12 [6].

Table 1 Mean and standard deviation of specimen properties.

Parameter	IM	SLSx	SLSz
Test diameter (mm)	7.10±0.05	7.49±0.11	7.41±0.14
Density (g/cm^3)	1.03±0.01	0.97±0.01	0.98±0.01
Ra-value (µm)	0.7±0.05	15±0.25	18.5±0.25
UTS (MPa)	53±1.4	52±1.5	49±1.7
E-modulus (MPa)	1701±45	2080±55	2158±46
ϵ-fracture (%)	97±3	7±0.8	4±0.3

Figure 3 shows the corresponding average tensile curves for the SLSx, SLSz and IM samples. These curves indicate very low fracture-strain values (4% and 7%) for the SLS parts compared to 97% for the IM parts. It is also clear that for quasi-static loading of SLS parts, interlayer fracture (between two successive layers) is more brittle than intralayer fracture (within the layers). In general, the tensile properties for all specimens are comparable to values reported in literature for PA12 (PA2200) [2,6].

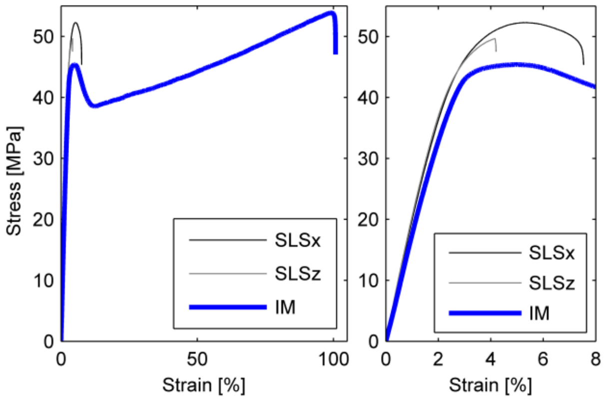

Figure 3. Tensile properties of SLSx, SLSz and IM parts in PA12 (PA2200), full scale (left) and detail for strain<8% (right).

3.2. Fatigue behaviour of plain specimens

Unnotched SLSx, SLSz, and IM specimens were tested at 3Hz with four different stress amplitudes. For each stress level at least three specimens were tested and stress-life (SN) curves were constructed. Figure 4 shows the SN-line for the SLSx samples on a semi-logarithmic scale. The data points of these samples are not displayed for improved readability. A clear fatigue limit is present. At 10^6 cycles, all specimens endure stress amplitudes of at least 18MPa, which is high compared to other semi-crystalline polymers [6].

Despite the distinct difference in ductility between samples produced by SLS and IM, and the clear anisotropy in fracture strain of the SLS parts, the SN curves indicate little variation in fatigue resistance between all tested samples (for test results and SN curves see [5]). For the plain SLSz and IM samples, the maximum deviation to the reference SLSx SN-curve in cyclic stress amplitude for a given number of cycles is limited to 6MPa in the low cycle range (10^0 - 10^4 cycles) and 4MPa in the high cycle range (10^4 - 10^6 cycles). This can be partially explained by the optimal process conditions that were used for selective laser sintering. Nearly all powder particles are fully molten, leading to low porosity (<5%), high ultimate tensile strength (>45MPa), and little variation in these properties between all samples as shown in Table 1. The similarity in fatigue resistance is also caused by a thermal effect that influences the failure process as explained in the following paragraphs.

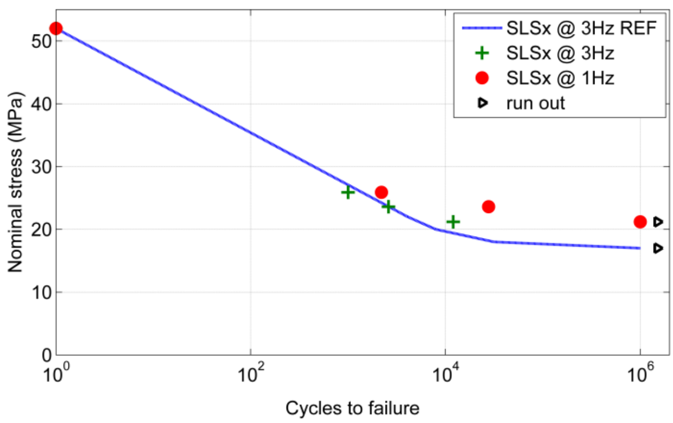

Figure 4. Fatigue properties of plain SLSx parts tested at 3Hz and 1Hz.

The semi-crystalline PA12 material is characterised by large internal damping and low thermal conductivity. Therefore, thermal heating occurs when the samples are tested under constant stress amplitude. Since no artificial cooling is applied, adiabatic conditions apply and hence the generated heat is transferred into temperature rise of the sample. This temperature rise during cyclic testing can be related to the area within the hysteresis curves [4].

Figure 5 presents such curves for a plain SLSx sample subjected to a load amplitude of 1kN with a test frequency of 1Hz. A progressive increase of the dissipated energy is noticed during testing. Initially, the temperature rise of the specimen does not affect its mechanical properties. After sufficient cycles, the temperature increases above the glass transition region of 23.5-55°C. As a result, the amorphous polymer chains of the semi-crystalline PA12 soften and reorient themselves and the stiffness of the material decreases, as indicated by the slope of the final hysteresis loop. This cyclic softening continues until the sample can no longer withstand the load and breaks. Since both SLS and IM parts have similar crystallinity values of 28% and 25% respectively [4], this process is not affected by the manufacturing method. In addition to the cyclic softening, a cyclic creep mechanism is active. The mean displacement of every cycle increases as the number of cycles increases, as shown in Figure 5.

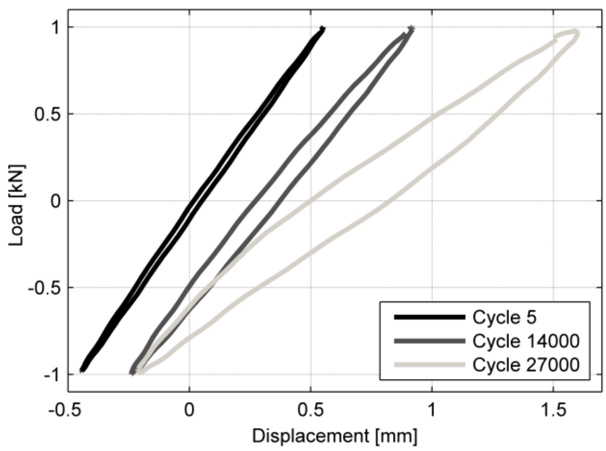

Figure 5. Hysteresis curves for a plain SLSx sample subjected to a load amplitude of 1kN at a test frequency of 1Hz.

When the combination of stress amplitude and test frequency is sufficiently low, the heat generated within the specimen is in equilibrium with the heat transferred to the surroundings. In that case, the fatigue resistance increases significantly when the temperature of the specimen stabilises below the glass transition region. This is illustrated in Figure 4, which shows the results of 6 additional experiments on plain SLSx specimens tested at 3Hz (+) and 1Hz (o) respectively. These samples were tested until failure with load amplitudes of 0.9kN, 1kN, and 1.1kN. The resulting lifetimes for the experiments at 3Hz coincide with the stress-life curve of the previous experiments as expected. The tests at 1Hz yield substantially longer lifetimes than the tests at 3Hz, ranging from 2.2 to more than 83 times longer for the same stress amplitude. The sample tested at 0.9kN and 1Hz showed no temperature accumulation and did not fail at 1.2×10^6 cycles.

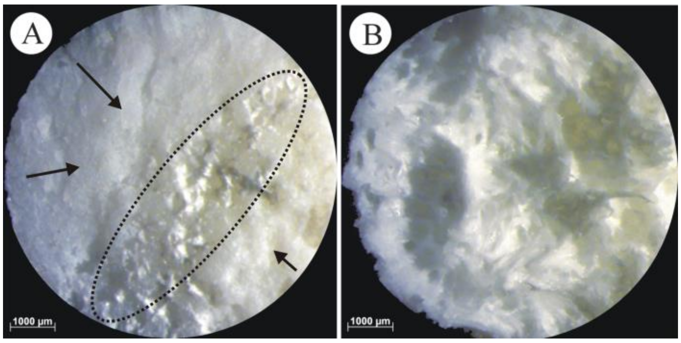

Figure 6. Fracture surfaces of plain SLSx samples subjected to load amplitudes of 1kN with test frequencies of 1Hz (A) and 3Hz (B).

Figure 6 shows the fracture surfaces of two plain SLSx samples that were tested at the same stress level but with different test frequencies. When tested at 1Hz (Figure 6A), the fatigue loading resulted in a brittle fracture perpendicular to the loading direction after 27000 load cycles. The surface temperature of this sample stayed below the glass transition region for ±14000 cycles. Within this period, the specimen did not soften and the high mechanical load caused cracks to initiate from unmolten powder particles inside the sample, as explained in [3], and to propagate as indicated by the arrows in Figure 6A. Almost no signs of plastic deformation can be distinguished. After continued loading, an increase in temperature above the glass transition region caused the stiffness to decrease as shown in Figure 5. This eventually led to the slightly more ductile overload fracture as indicated by the dotted ellipse in Figure 6A.

When tested at 3Hz (Figure 6B), the fatigue loading resulted in a ductile fracture after 2600 load cycles. Substantial plastic deformation is noticed as indicated by the semi-spherical depressions and drawn out peaks and fibrils. In this case, the temperature increased rapidly and the gradual softening of the material occurred almost instantly, leading to a much shorter life than the specimen tested at 1Hz. It should be noted that a slight increase in specimen temperature could in some cases also be beneficial for the fatigue life. The increased molecular mobility can cause the amorphous polymer chains to stretch and elongate in a preferential orientation leading to improved fatigue resistance. However, this is not the case for the specimens discussed here, since the progressive temperature rise clearly has a detrimental influence on the fatigue resistance.

In general, a clear difference in failure mechanism is noticed for plain SLSx specimens tested at equal stress amplitudes but different test frequencies. In both cases, however, the mechanical load resulted in fatigue striations similar to those shown in Figure 9E and more in detail in Figure 9F.

3.3. Fatigue behaviour of notched specimens

Notched SLSx, SLSz, and IM specimens were tested at four different stress amplitudes. For each stress level at least three specimens were tested and stress-life (SN) curves were constructed as shown in Figure 7 for the notched SLSx samples. In correspondence with the plain specimens, the variation in fatigue behaviour between SLSx, SLSz and IM parts is limited. However, the SN-curves of the notched samples indicate improved fatigue resistance with respect to the plain samples in the region between 10^0 and 10^5 load cycles. This effect weakens towards 10^5-10^6 cycles. The improved fatigue strength of the notched samples can be explained as follows:

(a) For quasi-static loading, a notch strengthening effect is active, i.e. an increase load carrying capacity of the notched area after local yielding. This is confirmed by tensile tests on the notched samples.

(b) For loading between 10^0-10^5 cycles, there is a reduced thermal load compared to the plain samples. Less heat is generated in the material since a smaller volume is subjected to cyclic deformation. This is confirmed by temperature measurements of the surface during testing. Also, the more brittle fracture surface of the notched samples (similar to Figure 6A) compared to the ductile fracture of the plain specimens (similar to Figure 6B) confirms the hypothesis that the failure of the notched parts is less influenced by thermal effects.

(c) For loading between 10^5-10^6 cycles, the stress amplitude is low and no progressive heating occurs. The failure mechanism for plain and notched samples is identical and only related to the mechanical load. Cracks nucleate from unmolten powder particles inside the specimen and hence the peak stress caused by the geometrical stress raiser does not influence the mechanical failure of the samples.

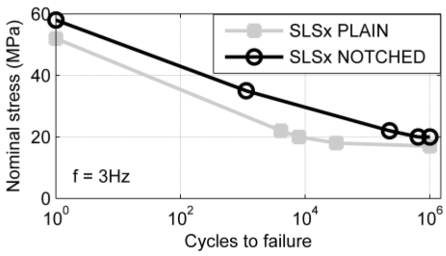

Figure 7. Fatigue properties of plain and notched SLSx parts in PA12.

To further analyse the fatigue properties of notched parts near the endurance limit, a CT-scan was taken from a notched SLSx sample (Figure 8). This scan shows remaining pores in the part acting as local stress raisers, causing crack nucleation. Because of the contour scanning that is applied after scanning of each layer, no pores are present near the surface, which further explains the fact that the notch does not have a detrimental effect on the fatigue life since failure initiates from pores within the specimens.

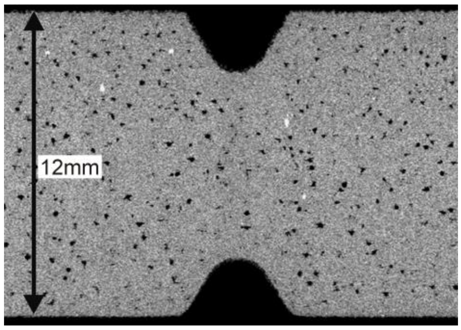

Figure 8. CT-image of notched SLSx sample.

3.4 Influence of the notch manufacturing method

The influence of the notch manufacturing method is studied by comparing fatigue properties of SLSx and SLSz parts in which a notch is mechanically machined with SLSx and SLSz parts that have an as-built notch. The machined notch was made using a special lathe bit for polymers. The notch geometry of these parts was carefully measured and these measurements were then used to generate the CAD-file for the laser sintered notch.

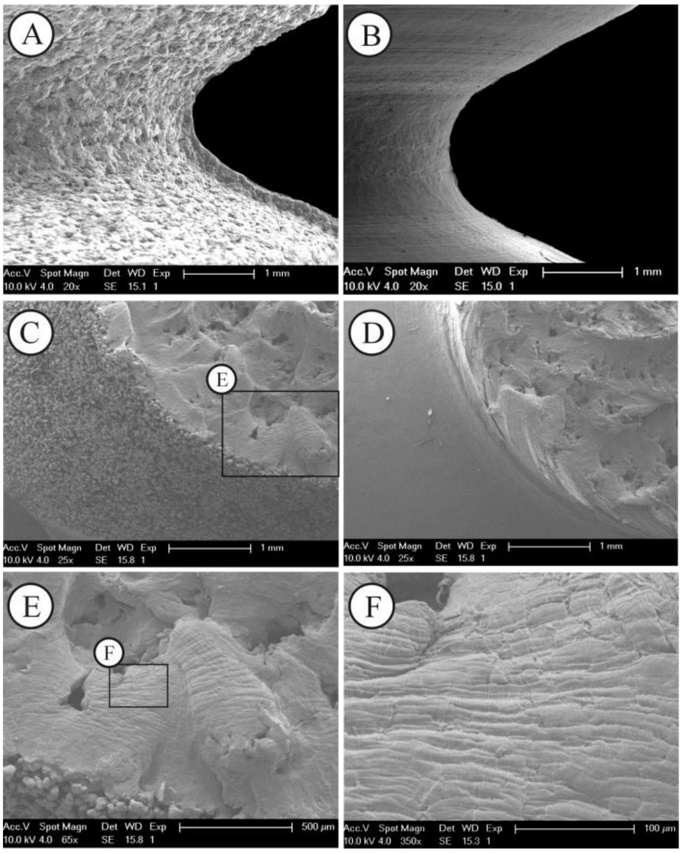

Figure 9. SEM images of notched SLSz parts: SLSed notch (A,C), mechanically machined notch (B,D), and fatigue striations (E,F).

Figure 9 presents scanning electron microscopy (SEM) images taken from as-built (A,C) and machined (B,D) notches of SLSz parts. All specimens were sputter coated with gold-platinum to achieve high contrast and to prevent damage to the microstructure.

Figure 9A and B clearly show the difference in surface roughness. For the as-built notch, the high surface roughness (Ra = 18.5μm) is caused by powder particles that are partially molten to the surface. These particles do not carry any load and therefore they do not act as stress raisers.

Figure 9C and D show a quarter segment of the fracture surface from fatigue tests with stress amplitude of 35MPa and test frequency of 3Hz. In both cases, fatigue striations are noted as shown more in detail in Figure 9E and F.

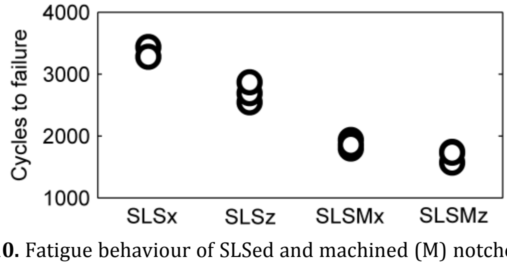

Figure 10. Fatigue behaviour of SLSed and machined (M) notches in SLSx and SLSz parts subjected to stress amplitudes of 35MPa at 3Hz.

Figure 10 shows the number of cycles to failure for the specimens with as-built (SLSx and SLSz) and machined (SLSMx and SLSMz) notches. For each of these specimen types, three samples were subjected to a 35MPa stress amplitude at 3Hz. The small variation in fatigue life for specimens of the same type is clearly visible. In addition, parts with as-built notch have a significantly improved fatigue resistance with respect to the parts with mechanically machined notch. This effect is probably caused by the absence of the strong outer material layer in the parts with machined notches. However, more work is needed to study this effect for different combinations of cyclic loads and frequencies.

4. Conclusions

In this work, the fatigue behaviour of plain and notched SLS and IM parts in PA12 was studied with special focus on the influence of the test frequency and the notch manufacturing method. This led to the following conclusions.

- The building orientation of the SLS specimens has no influence on the fatigue properties. This is due to the optimal production parameters that were used for the SLS process, leading to near full-dense parts with low variation in mechanical properties and equal intra- and interlayer fatigue strength. In addition, SLS specimens have the same fatigue properties as IM specimens produced from the same PA12.
- The test frequency significantly influences the fatigue life of SLSed PA12 parts. At certain combinations of stress amplitude and test frequency, cyclic softening occurs if the sample temperature reaches the glass transition region. This leads to reduced fatigue properties and ductile fracture.
- For a test frequency of 3Hz, the notched SLS samples show improved fatigue resistance compared to the plain SLS samples. This is caused by the reduced thermal load and by the strong outer material layer of SLS samples. Cracks nucleate from unmolten powder particles and these are not present on the surface since contour scanning is applied. Hence failure initiates from within the specimen, and the notch does not have a detrimental effect on the fatigue properties.

More work is needed to fully understand the transition between the mechanical and the thermo-mechanical failure mechanisms for any combination of stress amplitude and test frequency. In a next step, the developed methodology will be extended to other polymers that can be produced with SLS.

Acknowledgement

This research was supported through a Ph.D grant of the Institute for the Promotion of Innovation through Science and Technology in Flanders (IWT-Vlaanderen).

References

[1] Kruth, J.-P., Levy, G., Klocke, F., Childs, T.H.C., 2007, Consolidation phenomena in laser and powder-bed based layered manufacturing. CIRP Annals 56/2:730-759.

[2] Goodridge, R.D., Tuck, C.J., Hague, R.J.M., 2012, Laser sintering of polyamides and other polymers. Progress in Materials Science, 57:229-267.

[3] Van Hooreweder, B., De Coninck, F., Moens, D., Boonen, R., Sas, P., 2010, Microstructural characterisation of SLS-PA12 specimens under dynamic tension/compression excitation. Polymer Testing, 29/3:319-326.

[4] Van Hooreweder, B., Moens, D., Boonen, R., Kruth, J.-P., Sas, P., 2013, On the difference in material structure and fatigue properties of nylon specimens produced by injection moulding and selective laser sintering, Polymer Testing, 32/3:972-981.

[5] Van Hooreweder, B., Sas, P., Moens, D., Boonen, R., 2013, Development of accelerated multi-axial fatigue tests based on scaling laws. PhD thesis, KULeuven.

[6] Lampman, S., Bonnie Sanders, B., Hrivnak, N., Kinson, J., Polakowski, C., 2003, Characterization and Failure Analysis of Plastics. ASM International.