

Long-term Properties of Laser Sintered Parts of Polyamide 12

Influence of Storage Time and Temperature on the Aging Behavior

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D. Drummer¹, R. Harder¹, G. Witt², A. Wegner², K. Wudy¹, M. Drexler¹

¹ University of Erlangen-Nürnberg, Erlangen, Germany

² University of Duisburg-Essen, Duisburg, Germany

Abstract—The key in selective laser sintering (SLS) of polyamide 12 for exceeding the cusp from a prototyping to a serial production technique poses the long-term behavior of such components. The improvement of additive manufacturing technologies is attended by the enlargement of the possible ranges of use. The crucial obstacle for their actual use is the ignorance of the long-term properties and the consequential fact of not given warranty. The aging behavior of polyamide 12 based injection molded parts and powder for the SLS process is largely probed and methods of analysis are well-established. On the basis of several build jobs consisting of vertically build tensile bars, oven storage experiments are realized. Storing for several time periods and temperatures allows a first calculation of the aging behavior in consideration of the building position and process parameters of each bar. First of all the adaptability of two methods of analysis, tensile test und solution viscosimetry, for determining the state of the aging of SLS parts is surveyed. It shows that depending on the used method of analysis the building position of the picked up sample could play a role. Furthermore the influence of the process parameters, the aging processes itself and the necessary storage conditions for the artificial aging of the parts are considered. All in all this article gives a review of the aging mechanisms of oven stored polyamide 12 parts, possible methods of analysis to determinate their state of aging and first results to qualify the adaptability to selective laser sintered parts. In Addition to that a first correlation between mechanical and chemical properties is made. The presented results form a first step for the future to be able to guarantee the requested product quality over its lifetime, but nevertheless other methods of analysis have to be qualified for the determination of the aging behavior of SLS parts.

Index Terms—additive manufacturing, selective laser sintering, polyamide 12, long-term properties, aging behavior, thermo-oxidative degradation

I. INTRODUCTION

The additive manufacturing technique “Selective Laser Sintering” (SLS) established itself in several areas of application. Beside the utilization in the design or art sector and the conceptional modelling, the SLS process is used in the prototype construction [1, 2]. Though the aim is the additive manufacture of functional and mechanically loadable parts, also in (small) batch production.

There are already a few applications, such as a Fin-Gripper¹ or a ventilation duct² for laminar air flow which show the great potential of this manufacture technique. One of the reasons why there are only so few applications is the ignorance about the long-term properties of laser sintered polyamide 12 parts.

Albeit there are findings on the aging behavior of injection molded parts and polyamide 12 (PA12) powder during its manufacture the question arises, if this knowledge is applicable [3, 4]. This includes beside the aging mechanisms itself the analysis methods to determine the state of aging and the specimen sampling, where the dependence on the building position has to be considered [5, 6].

II. FUNDAMENTALS

A. Aging of polyamide 12

To initialize aging processes there has to be at least one cause. The possible cause can be classified in two groups, the inner and the outer aging causes. Examples for outer aging processes are mechanical and thermal loads, the exposure of diverse media particularly oxygen or radiation [7, 8]. Causes as residual stresses as a result of e.g. inhomogeneous cooling or orientations belong to the group of inner aging causes. Another can also be only partial proceeded chemical reactions, e.g. the post condensation reaction, which might apply to the production process of a commonly used polyamide 12 for the selective laser sintering process [7].

If there are aging causes aging processes can start. These processes can be subdivided, too. It is spoken of physical and chemical aging processes. The physical aging processes include predominant reversible changes of the crystalline structure or the external shape. Examples are the relaxation of orientations or the post-crystallization [5]. Contrary to them are the chemical aging processes, which distinguish itself by irreversible changes of chemical bonds. To this category belong e.g. changes on the chemical composition or macromolecular structure. A very important respectively the probably most relevant chemical aging process under oxygen atmosphere is the thermo-oxidative degradation, where macromolecule chains are cracked under the external hazard of temperature and oxygen. Schnabel describes in [9] the auto oxidation of polymers which is a radical chain mechanism. Under the effect of temperature radicals are generated

¹ Festo AG & Co. KG, Ostfildern, Germany

² EOS GmbH, Krailing, Germany

which initiate the auto oxidation process and react with oxygen [7, 10].

The described aging processes manifest itself in several aging phenomena. To this rank among manifold property changes, concerning mechanical, chemical or optical changes. Furthermore the thermo-oxidative degradation can cause discolourations. As a consequence of the post crystallization post shrinkage could appear [7].

B. Determining factors on the part aging

The occurrence of the part aging depends beside the component properties on the component loads. The component properties are affected by the properties of the used raw material for the SLS process as the flowability or the thermal prehistory, which, in turn, have an impact on the production process in combination with e.g. the process parameters. This gives rise to raw material and process dependent part properties, such as physical structure (crystallinity or orientations), chemical composition (molecular weight and its distribution or branching grade) or macroscopic properties (surface texture or porosity). The connection between the used production process and the resulting part properties is considered below [3, 10].

The SLS process has got characteristic process steps and possesses respective attributes by what the resulting part properties are affected. The processes can be described with three single process steps. These are the coating, energy input and material consolidation for the SLS process. The energy input by the laser causes no remarkable dynamic in the melt-pool so there is merely innate melt dynamic existent. In consequence of that an orientation of macromolecule chains should not be induced. In the SLS process there is no process pressure in the powder bed. This explains the rough surfaces and the comparatively high shrinkage and porosity of SLS parts. As a result of the slow cooling of the SLS parts they might have a less affection to internal stresses. In addition to that the cooling conditions also determine the crystalline forming what could have as orientations an effect on e.g. the gas diffusion. Generally the thermal loading of the polymer during the process is a matter of consequences. Because of the build-up in layers the lower layer get thermally higher loaded than layers at the end of the buildjob. This might cause differing predegradations of the polymer along the buildup direction. Another irregularity within the part properties could be caused by an inhomogeneous temperature distribution across the powder bed surface [5, 3, 8, 11, 12].

III. MOTIVATION

As mentioned in the introduction, the transferability of findings concerning the aging behavior of injection molded parts to SLS parts could be constricted. This is getting confirmed having a closer look at the injection molding process. As a consequence of the injection step the macromolecule chains could become oriented, the highly appearing pressures in the metal cavity could counteract the shrinkage and porosity and cause defined surface properties. Generally injection molded parts cool down comparatively fast what could influence the morphological structure e.g. the crystallinity [13].

Consequently different processes and their parameters can produce different part properties and you can assume that there might be a process specific aging behavior which is only restricted transferable. Hence investigations

concerning the influence of different process parameters, variation of the storage time and temperature in circulation oven storage experiments on the aging behavior of laser sintered polyamide 12 parts are performed.

IV. MATERIAL AND METHODS

A. Material

Within these experiments a refreshed polyamide 12 (PA12) powder is used. The powder PA2200 of the company EOS GmbH based in Krailling, Germany, had been refreshed by using the recommended constant strategy of adding 50 per cent by weight to the left over powder of previous SLS buildjobs. Its Melt-Volume-Rate (MVR), measured at 235 °C with a load of 5 kg is 26,3 cm³/10min. This value had been measured according to DIN EN ISO 1133 at the Chair for Manufacturing Technology of the university of Duisburg-Essen. It is an indicator for the material quality and decreases with the powder age and is a proportion for the average molecular weight, which increases, and its distribution [14, 15]. The MVR for new PA2200 powder measured at the same conditions is varying depending on the used batch. The values diversify form a shade above 60 cm³/10min up to a bit more than 80 cm³/10min [16].

B. Specimen production and sampling

a) Specimen production

By means of two build jobs 220 tensile bars were built in upright z-position (Fig. 1). Each job, shown in Fig. 1, consists of two times 55 bars which were manufactured with standard respectively optimized parameters next to each other as given below. The used machine is an EOS Formiga P100 of the company EOS GmbH based in Krailling, Germany. The coating occurs in x-direction and the build job took about 13 hour and ten minutes at a height of 163 mm.

The used optimized process parameter set is based on the recommended process parameter set of the company EOS GmbH based in Krailling, Germany for the manufacture of PA2200. Wegner presents it in [17] as a result of the optimization in consideration of “[...] good mechanical properties, high part density as well as high accuracy” [17]. Thereby the laser power, hatch distance and scan speed were systematically varied (cf. table I) to “[...] establish a robust parameter set being less sensitive to process variations by material quality a cooling conditions as well as temperature distribution” [17].

TABLE I.
RANGE OF VARIED PROCESS PARAMETERS [17]

Process parameter	Range
Laser power [W]	20 ... 25
Hatch distance [mm]	0.15 ... 0.2
Scan speed [mm/s]	3300 ... 4500

Out of the varied parameters results a volume energy density from 0.22 up to 0.51 J/mm³. “The limits were chosen on the one hand by means of the results of a former study (lower limit) and on the other hand to reach polymer degradation [17].

b) Speciem Sampling

The known and described above fact of the possibly varying part properties according to the part build position it necessitates its observance. In case of an inhomogeneous temperature distribution across the powder bed surface, especially the temperatures in the corners of the build chamber could differ itself. Consequently the build chamber respectively the sampling area of each build job (cf. dashed line frame in Fig. 1) is subdivided in five areas as shown in Fig. 2). The arrangement of the five areas happened in consideration of several conditions. First there should be a preferably constant distance between the inner area (number 5) and the frame of the build job, secondly each corner area should imply at least one tensile bar per process parameter set and planned conditioning state. Out of the inner area the sampling of an additional retain sample has to be guaranteed.

To prevent the influence of the building position the sampling is made systematically distributed over the build chamber respectively the sampling area. If this effect is to be considered or the build position is inconsiderable, the sampling happens locally in one of the subareas.

C. Speciem conditioning and preparation

a) Speciem conditioning

The aim referred to the conditioning is the achievement of defined states. In this case defined states mean temporally limited thermal and oxidative load. Hence the tensile bars are stored in circulating atmospheric air ovens at 80/100/110/120/140 °C for several time periods of one, two and four weeks (cf. table II). The storage occurred vertically hanging to ensure homogeneous thermal condition across the part surfaces and between them. For this purpose a hole was drilled through the lower clamping surface (cf. Fig. 3). The upper and lower storage temperature limits were chosen on the basis of bibliographical reference for thermal short- and long-time limitations of use [18, 8].

TABLE II.
STORAGE CONDITIONS – VARIATION OF STORAGE TIME AND TEMPERATURE

Storage time	Storage Temperature in °C				
	80	100	110	120	140
1 week	X	X	X	X	X
2 weeks					X
4 weeks					X

Due to the moisture absorption of polyamide 12 and the well-known associated changes of the mechanical properties [8, 19] a reference state is defined. To this tensile bars were stored in an evacuated oven at 70 °C for two weeks until constant weight so that they were dried to the exclusion of oxygen. The residual moisture content after the conditioning of the reference state and the three lowest temperatures (80/100/110 °C) after a storage time of one week is below 0,15 per cent by weight measured using the Karl-Fischer-Titration.

b) Speciem preparation

Beside the tensile tests viscometric analysis were made. For the consideration of the above-mentioned build position dependency the sampling was carried out partially at

three positions (cf. Fig. 3) of one tensile bar through what the influence of the build job height can be considered. Due to the fact that the aging starts at the surface and proceeds towards the core [3] the samples were picked across the whole cross-section to equalize potential differences.

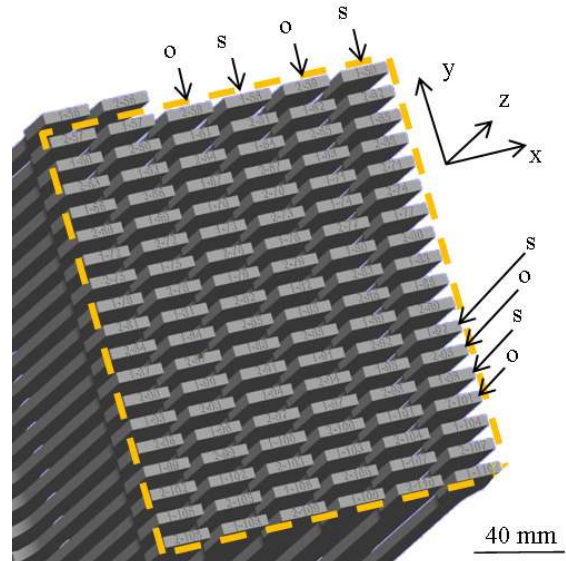


Figure 1. Build job consisting of two times 55 tensile bars (DIN EN ISO 3167, type A) with a length of 150 mm which were manufactured using standard (s) respectively optimised (o) parameters next to each other ; build direction: z-axis; coation direction: x-axis; Sampling area framed with dashed line

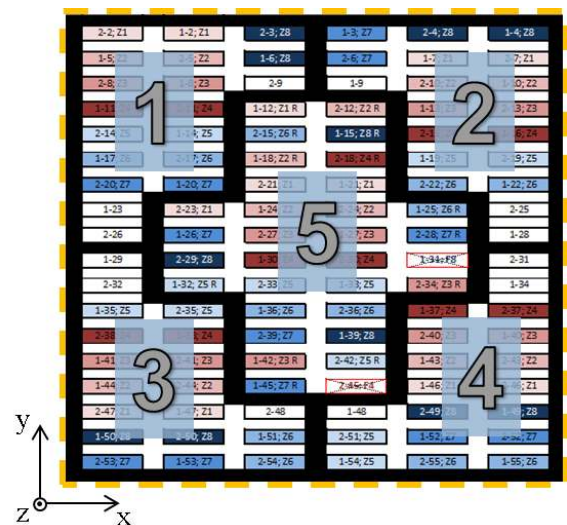


Figure 2. Subdivided speciem sampling area

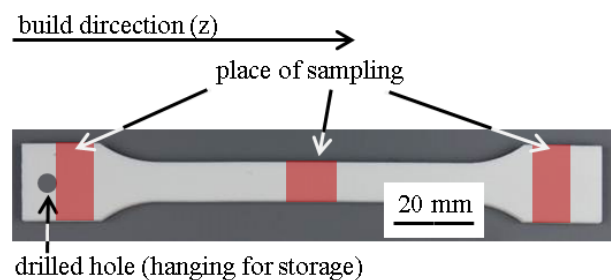


Figure 3. Specimen preparation for the procedure of the solution viscosimetry

D. Analysis methods

To analyze the mechanical properties in dependence on the conditioning state the mechanical testing of the tensile bars was carried out according to DIN EN ISO 527. The Young's modulus had been evaluated at a testing speed of 1 mm/min, the maximum stress and the elongation at break with a speed of 5 mm/min. The speeds were chosen so slow due to the as expected low measured forces [11, 20].

As the Melt-Volume-Rate the viscosity number is associated with the average molecule weight and its distribution [21]. Thus changes concerning the macromolecular structure could be detected. According to DIN EN ISO 307 the viscosity number was identified using the solution viscosimetry. Because of health issues here concentrated acid sulphur was used at 25 °C instead of m-cresol. An advantage of this analysis method is the little necessary specimen weight. So it is possible to consider the above-mentioned influence of the build position of the samples also over their length respectively height. The samples were picked out of the five subareas across the build platform and at three heights as shown in Fig. 2 and Fig. 3. For further examinations the specimen were sampled out of the middle section of the tensile bar which was built in the fifth (mid) subarea.

For the documentation of aging phenomena as discoloration pictures of representative parts had been taken.

V. RESULTS AND DISCUSSION

A. Influence of the build position on the viscosity number

As mentioned in the fundamentals based on further done studies correlations between build position and mechanical part properties are well known. This influence hadn't been analyzed concerning the viscosity number but might play a role because of the above-mentioned differing thermal loads on the polymer. Hence firstly the viscosity number is considered space-resolved. In Fig. 4 the viscosity numbers of 15 samples picked across the whole build chamber are diagrammed. Five samples in one particular x-y-layer across three heights as shown in Fig. 2 and Fig. 3. The specimens are unconditioned that means they also weren't dried to reach the reference state and consequently also weren't thermally loaded. For the purpose of comparison the viscosity number of new PA2200 powder is given which is about 60 ml/g [15]. The results of one tensile bar are presented with three bars side by side in the diagram. Thus any three-tuple arises out of one subarea. Though the calculated standard deviations are very small, the results indicate no considerable differences between the measured viscosity numbers. Herefrom the conclusion that one sample is representative for the whole build chamber can be drawn.

Consequently the sampling is carried out at only one position in the middle of the build job to analyze the influence of the storage time and temperature in the continuative storage experiments. For the following diagrams the viscosity number is only being determined by means of specimens picked out of the center of the build job. In this regard this method of analysis suits itself to detect the viscosity number independent of the sampling position.

B. Influence of the storage temperature

Fig. 5 shows the Young's modulus as a function of the storage temperature at a storage time of one week for each

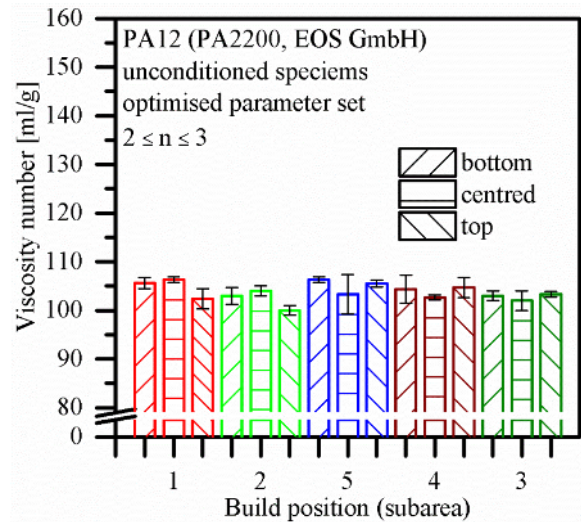


Figure 4. Space-resolved examination of the viscosity number

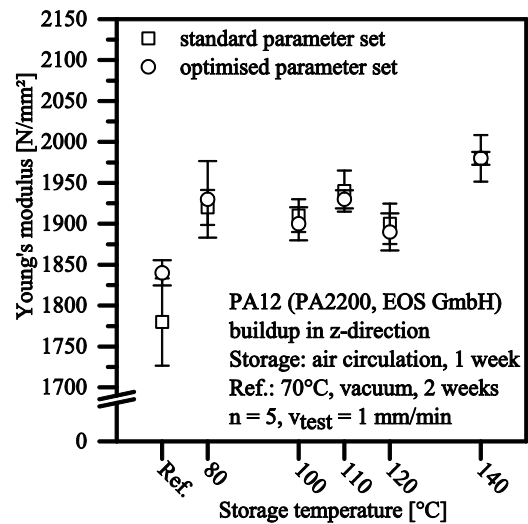


Figure 5. Young's modulus as a function of storage temperature

parameter set. Also the results for the referring charge are diagrammed. Over all storage temperatures there is no circumscribable difference between the two parameter sets. Solely the mean stiffness of the parts manufactured with the optimized parameter set tends to be slightly higher whereupon the scopes of the standard deviations overlap itself. As a result of the storage at 80 °C for one week the Young's modulus is increases. Further temperature increases up to 120 °C cause no effect, a storage at 140 °C provokes another increase of the Young's modulus.

A reason for the increase of the Young's modulus could be an embrittlement as a result of physical aging processes. Here the thermal load on the polymer might cause a change in its morphology. Consequently it could manifest itself in a decrease of the elongation of break and or prospective microscopic analysis. If these effects are only caused by physical aging processes the viscosity number should be constant.

The elongation at break as a function of storage temperature and the reference is figured in Fig. 6. Clearly to see are the large amounts of the calculated standard deviations which complicate a classification of the mean values against each other. Merely a tendency respectively differ-

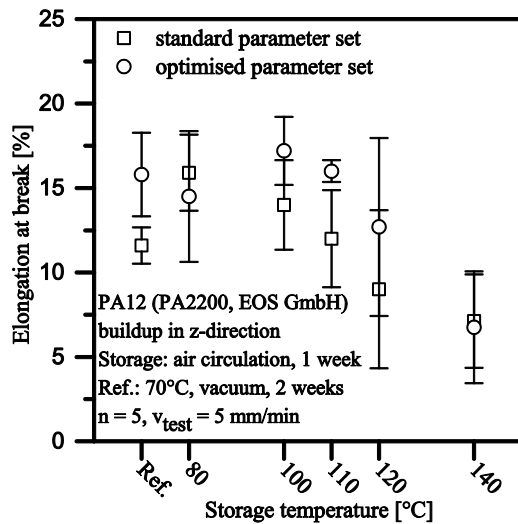


Figure 6. Elongation at break as a function of storage temperature

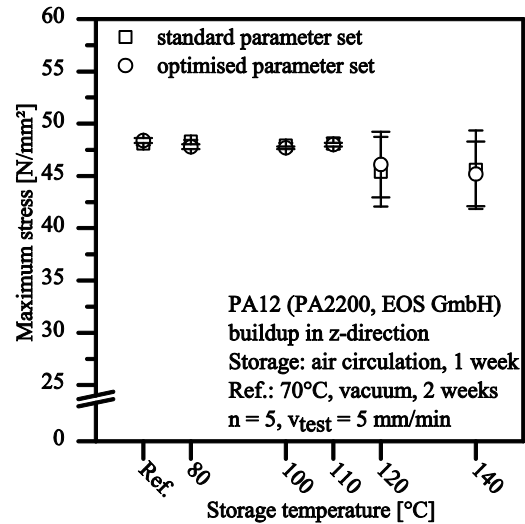


Figure 7. Maximum stress as a function of storage temperature

ence can be derived. The optimized parameter set might cause parts with a potentially higher elongation at break, albeit the difference equalizes latest at 140 °C and the elongations generally decrease. Similar to the Young's modulus the only tolerably delimitable results are those at 140 °C which show a more obvious drop.

The above-mentioned drop of the elongation at break at 140 °C fits to the increase of the Young's modulus at the same temperature. If this is a reason of thermo-oxidative degradation a decrease of the viscosity number should be seen as shown e.g. in [3]. The viewing on the change of the viscosity number is made below. Another reason or a superposition of different ones for the shorter elongation could be an earlier failure of the tensile bars as a result of e.g. surface embrittlement or the notch effect of the laser-sinter typical rough part surface. A general problem by analyzing the elongation at break for laser sintered tensile bars is the comparatively low initial value which is much higher for injection molded unreinforced polyamide 12 parts³.

The examination of the maximum stress as a function of the storage time, as shown in Fig. 7, results that there are no evaluable differences. Merely the storage at 120 and 140 °C tends to higher calculated standard deviations and a slight drop.

This slight decrease and the larger deviations could have several reasons. On the one hand the above-mentioned embrittlement with possibly involving craze formation could be one in consequence of which a dispersive material failure appears. On the other hand variability of the part porosity could also justify the larger standard deviations. The background therefore is that with an increasing porosity the load-bearing cross-section decreases and the failure could occur at the weakest position. Beside the part porosity itself, embedded unmelted powder particles or a locally higher porosity what could cause tension peaks might also result in an extraordinary material failure.

Beside the mean values of the five storage temperatures and the reference state in Fig. 8 the mean viscosity number of the unconditioned samples is diagrammed. As a

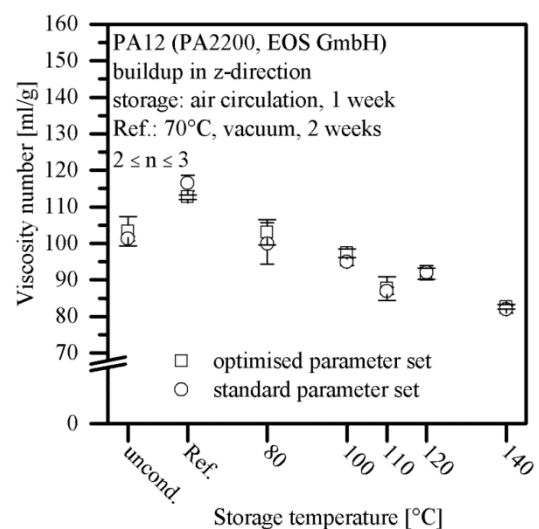


Figure 8. Viscosity number as a function of storage time

consequence of the drying step the viscosity number increases compared to the result of the unconditioned state. The storage at 80 °C causes no changes in comparison to the unconditioned one. A further temperature increase affects a potentially decrement of the viscosity number. A difference between the standard an optimized parameter set is not ascertainable.

The decrease of the viscosity number could be an indication for thermo-oxidative aging what also could express itself in a change of colors of the artificial aged part. Accordingly as mentioned above the increase of the Young's modulus couldn't only be caused by physical aging processes. The fact that the viscosity number increases because of a storage at 70 °C in the vacuum and seems to stay unchanged after a storage at 80 °C in an air circulation oven allows the assumption that there are opposing trends during simultaneous exposure of temperature an oxygen. Here possibilities could be a post condensation polymerization working against a thermo-oxidative degradation of macromolecule chains [10, 22]. A direct comparison between these two values unfortunately isn't possible because of the different storage times. In [3] an increase of the viscosity number of a polyamide 66 sample as a result of e.g. post condensation polymerization after a

³Grilamid L 20 G, EMS-GRIVORY, EMS-Chemie AG

storage at 140 °C is termed, what could match with the behavior of the laser sintered polyamide 12 parts. Another interesting fact ascertained on injection molded polyamide 66 parts is that the viscosity number at the surface increases while the value in the center of the sample behaves itself as stored in an inert atmosphere. The reason therefore is the limited diffusion of oxygen. If this is transferable to the porose laser sintered parts is open yet.

In regard to the fact that there are no noteworthy differences between process parameter sets the conclusion could be drawn that the polymer doesn't get augmented thermally loaded using the standard parameter set compared to the optimized one.

Viewing at the artificial aged part in Fig. 9 it can be seen that a clear change of color can first be detected at temperatures above 120 °C. As mentioned in the fundamentals the discoloration can be a consequence of thermo-oxidative degradation. Combined with the results of the solution viscosimetry which say that there is a reduction of the average molecule weight after the oven storage at higher temperatures, the conclusion that the thermo-oxidative degradation first could have a detectable effect at higher temperature at a storage time of only one week is enabled.

C. Influence of the storage time

The increase of the storage time at a temperature of 140 °C causes no major differences regarding the Young's modulus in Fig. 10. Alike no influence of the parameter set is clearly detectable.

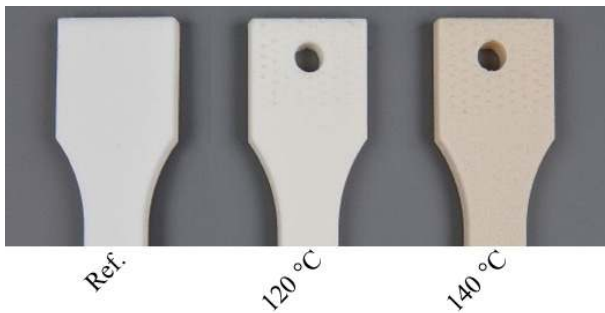


Figure 9. Change of colour as a consequence of storage at higher temperatures for one week

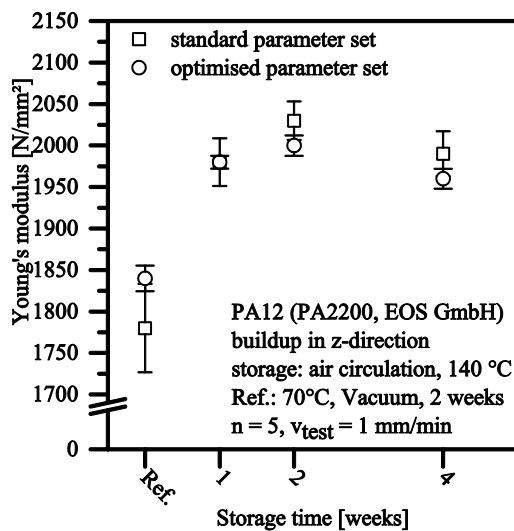


Figure 10. Young's modulus as a function of storage time

Conflicting to the Young's modulus the elongation at break, in Fig. 11, drops with the increase of the storage time. A better differentiation of the mean values though is only possible between the storage time of one and four weeks. Therefore it is difficult to say whether there is e.g. an ongoing embrittlement, a change in the morphology/crystallinity or the values seem to stagnate as a reason of the superposition of contrary changes.

The behavior of the Young's modulus argues that the embrittlement doesn't proceed. Therefore the elongation at break should stay as well unchanged.

The viewing on the maximum stress as a function of storage time shows in Fig. 12 that there is no clear discrepancy among the two process parameter sets. A better differentiation of the mean values though is only possible between the storage time of one and four weeks in which the value for the four-week one is smaller. It could be said that the tensile bars fail earlier with increasing storage time. This assumption could be confirmed with the result of the analysis of the elongation at break in Fig. 11, where a decrease of the elongation is diagrammed.

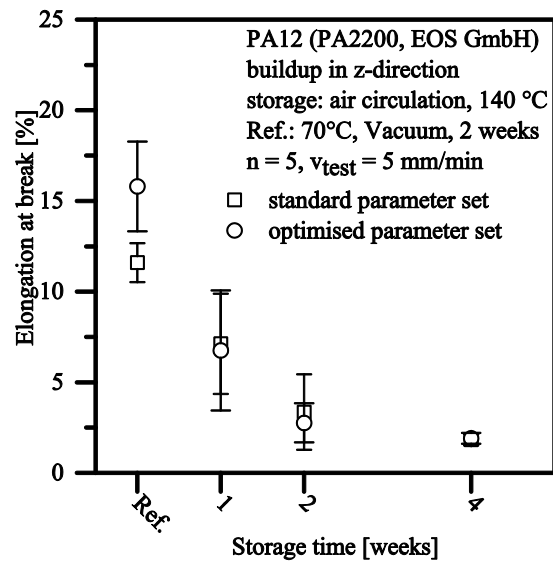


Figure 11. Elongation at break as a function of storage time

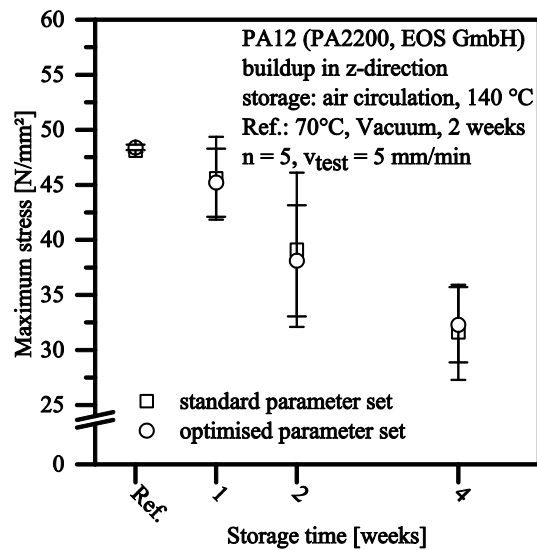


Figure 12. Maximum stress as a function of storage time

In Fig. 13 the viscosity numbers for the three storage times, the reference and unconditioned state are diagrammed. The extension of the storage time at a temperature of 140 °C causes no further remarkable decrease of the viscosity number. Furthermore there is no difference between parts which were manufactured with different parameters.

The extension of the storage time accompanies to an ongoing color change as shown in Fig. 14. This change could be caused by the thermo-oxidative degradation.

As already mentioned the thermo-oxidative degradation causes a decrease of the average molecule weight for polyamides what expresses itself on a drop of the viscosity number. The examination in regard to the discoloration argues for the thermo-oxidative degradation whereas the decrease of the viscosity number seems to stagnate at longer storage times. An explanation could be the limitation of the oxygen diffusion. While the surface is getting degraded the core of the sample stays unattended and the less degraded inner polymer predominantly influences the results of the solution viscosimetry.

VI. CONCLUSION

Beside the aging behavior itself the adaptability of the used methods of analysis to determine the state of aging is a in this article considered point. It is shown that for the application of the solution viscosimetry the sampling can be carried out location-independent. Like for injection molded tensile bars especially the elongation at break is susceptible to aging as well as the viscosity number.

The mainly drawable conclusion concerning the aging behavior is that there is a decrease of the mechanical properties and polymer degradation as results of manifold processes and their interactions between them. The parallel but contrary occurring processes post cross-sectioning and thermo-oxidative degradation show their influence viewing the results of the viscosity number and the devolution of the discoloration. A storage in a vacuum at 70 °C for two weeks causes an increase of the viscosity number whereas a storage in an air convention oven at 80 °C for merely one week cause no changes compared to unconditioned samples. Furthermore a doubling of the storage time from two to four weeks in an air circulating oven at 140 °C cause no further remarkable changes of the viscosity number whereas the discoloration proceeds. This could confirm the results form artificial aged injection molded polyamide parts where this effect is attributed to the limitation of oxygen diffusion. Changes concerning the morphological structure couldn't be qualified subject to storage time or temperature.

Hence the process parameters were optimized concerning mechanical properties an improvement of the aging couldn't be ascertained. One of the above-given reasons for the existence of lager calculated standard deviations is the part porosity and the consequently smaller load-bearing cross-section. For this reason the results of the mechanical testing have to be related to the part porosity. Additionally the influence of the part surface, concerning the notch effect, has to be considered. Also possible changes of the morphology are going to be surveyed. Another interesting detection which should be investigated more detailed is the limitation of oxygen diffusion and the potentially slower progression of thermo-oxidative degradation in the core of the sample. Therefore a storage

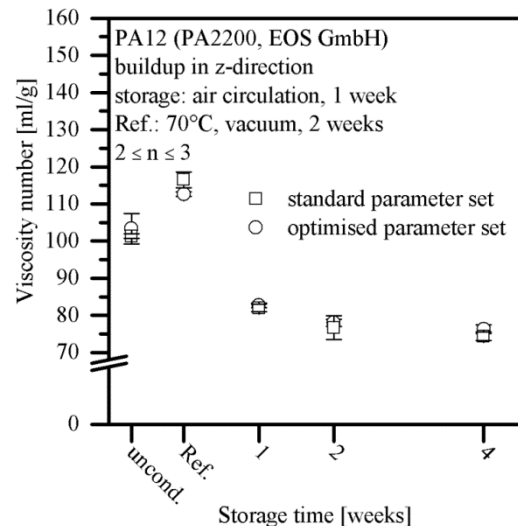


Figure 13. Viscosity number as a function of storage time



Figure 14. Colour change as a consequence of artificial aging

in an inert atmosphere or infrared spectroscopic measurements across the cross-section might be useful. Generally the within this paper presented work will be pursued on a larger scale. This implies the conditioning of more samples also for longer time periods. Regarding to the relevance to practical applications a testing of the material at common environmental conditions will be indispensable.

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AUTHORS

D. Drummer is with Institute of Polymer Technology, University of Erlangen-Nürnberg, 91058 Germany (e-mail: info@lkt.uni-erlangen.de).

R. Harder is with Institute of Polymer Technology, University of Erlangen-Nürnberg, 91058 Germany (e-mail: harder@lkt.uni-erlangen.de).

G. Witt is with Chair of Manufacturing Technology, University of Duisburg-Essen, 47057 Germany (e-mail: gerd.witt@uni-due.de).

A. Wegner is with Chair of Manufacturing Technology, University of Duisburg-Essen, 47057 Germany (e-mail: andreas.wegner@uni-due.de).

K. Wudy is with Institute of Polymer Technology, University of Erlangen-Nürnberg, 91058 Germany (e-mail: wudy@lkt.uni-erlangen.de).

M. Drexler is with Institute of Polymer Technology, University of Erlangen-Nürnberg, 91058 Germany (e-mail: drexler@lkt.uni-erlangen.de).

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