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Review of “Deformation and cavitation at the spherulite scale of an isotactic polypropylene”

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Review of version 1

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Authors

The authors would like to thank the referee and the editor for the detailed and valuable comments on the manuscript. The comments and suggestions were helpful to improve the readability of the paper. Taking all these remarks of the referee into account, the present document aims at answering to the questions.

Reviewer 1 (Luca ANDENA)

Reviewer

The paper proposes a novel method of studying deformation of a semicrystalline polymer at the spherulite scale. The research is very interesting, with some exceptionally good results which would provide a valuable addition to the knowledge of this topic. However, the manuscript has several shortcomings which in my opinion need to be addressed before it is suitable for publication.

As a general comment, the language needs to be improved. There are many example of typos, errors and often improper use of some words which result in obscure parts of the text which the reader struggles with. I will list some of them later on, but clarity and accuracy of the text should be improved overall in my opinion.

Authors

The authors started by correcting the misspelled words due to miscellaneous and indicated as minor revisions. In the revised manuscript, there is no change in the text color (in black) for these corrections. In addition, the revised manuscript was also checked by a native English colleague before the re-submission.

Reviewer

The biggest point that needs to be addressed is also related to clarity, but in a different way. The

basis for this very interesting research are the synchrotron images, exceptional but by the author's admission limited in terms of resolution. I am convinced that the authors spent quite some time working on them, comparing them with previous data reported in the literature, and interpreting them. This is evident from the Discussion section, but the reader is quite helpless as she/he is forced to "see" things the authors write about in the images, in most cases with very little or no guidance. The authors assume that a reader will be able to correctly recognize the features described in the paper from the images which still struggle to resolve the structures at such incredibly high magnifications. This makes it all very hard to follow, and could be significantly improved by providing a few sketches and highlighting said features in the images, as proposed below with reference to sections 4.1-4.2-4.3.

Authors The authors admit that the approach was not well explained. The reviewer is right that much effort has been made in the recognition of the spherulites boundaries. An additional figure (now fig.1) was proposed and commented in subsection 2.1. As requested by the reviewer, sketches were drawn to better explain the idealized image of a voided spherulite. Moreover the figures corresponding to subsections 4.1-4.2-4.3 were annotated and the text was strongly modified.

Reviewer Additionally, some statements presented in the last part of the manuscript are in my opinion not supported by enough evidence, and again the reader is forced to believe the authors (as we were forced to believe seeing features which we're not sure they're exactly there), who should instead substantiate a little more some of their conclusions.

Authors As mentioned before, initially, no annotation was provided in some key figures. Indeed, the authors considered that the text was clear enough to describe what they had in mind, hoping this should lead (not forced) the readers to the same conclusions. Unfortunately, this was not the case. This is the reason why the authors took their time to modify various sections/figures of the revised manuscript.

Reviewer At the end of the paper I have an important comment that in my opinion the authors should address. Obviously, when we approach a scale at which matter is no more homogeneous (e.g. going inside spherulites), we can't expect continuum mechanics to work. What is the purpose of applying analytical tools and concepts derived from continuum mechanics (such as strain and stress tensors) in a context in which the essence of the mechanical behaviour is determined by features which clearly put us in a situation in which matter cannot be treated as a continuum anymore? It is not clear to me where the authors want to go. Is the goal to obtain some kind of mesoscopic, homogenized model which is built according to the structure at the microscopic level? While the investigation is extremely interesting from the material science point of view, I can't see how we can use the results from an engineering perspective and the whole 5.2 paragraph is quite confusing to me. If the authors want to keep it, I think they should provide better insight as to what the connection is between the micro and macroscale.

Authors Section 3.2 was dedicated to introduce the Volumes Of Interest (VOI). They are related to the Representative Volume Element (RVE) required for Continuum Mechanics concepts. What is discussed in the paper is the homogenized response of an average spherulite. This latter is then assumed to be an equivalent homogenized medium. The approach is not supposed to go inside the spherulite, down to the amorphous/crystalline lamellae. For engineering perspective, the most important message of the paper is that as soon as necking appears at the surface, a gradient of stress and strain is set through the three principal direction of the specimen. The engineering stress and strain are meaningless therefore.

Reviewer More in detail: p.1, L13: non linearity for PP is likely to occur WAY earlier than necking or any kind of plasticization, while still being in the (mechanically) reversible regime!

Authors In the introduction, the authors mentioned phenomena reported in the literature (references herein). It has been checked that no comment about reversible/irreversible regime was set.

Reviewer p.1, L27 and other places as well: the term "learning" is used improperly. What is "learning" of 3D

patterns? They can be detected, identified, analyzed... they can't be "learned". Same later for the images of section 3.3 which I don't think will be "learned", but "analyzed".

Authors The authors agree to change the word "learning" as suggested by the reviewer. However, in sections 4.1-4.2-4.3, there was really a long stage of learning of the patterns allowing the recognition of the boundaries of the deformed spherulites. As mentioned above, these sections were modified so as to better explain the approach allowing these boundaries to be drawn.

Reviewer Another example comes later on with the many instances of "viewgraph", which is an unusual word that the authors use as a synonym of "image", "figure" or "picture". Confusing. A viewgraph is "A graph (or similar chart) produced as a transparency for use with an overhead projector." and I don't think that's the case.

Authors The authors agree. Any occurrence of the word viewgraph was changed

Reviewer p.2: "clearly related to the ideally dense matrix, that is, responding to the deviatoric part of the stress and strain tensor" What does this sentence mean? The way it's written, I don't understand. I can guess, but the reader should not guess, she/he should receive info in a clear way

Authors The model, assumed by the authors of the paper to be intrinsic, does not account for any relationship between the hydrostatic pressure and the volume variation. This means that the medium is supposed to be dense. This term is used in the literature for metallic material, where void growth can be neglected. Plasticity theory using incompressibility and Von Mises (shear stress criterion) is then applied. The term "ideally dense matrix" was modified into "matrix with no void".

Reviewer Same goes for "voltropy", which I don't think is a real word. I can't find any reference or definition for it. If the authors would like to define a new term, they should do it clearly at the beginning, hoping it catches on. But I would rather call it isotropy in the volume, so that everybody clearly knows what we are talking about.

Authors The authors acknowledge awkward global search/replace "iso" to "vol". This term was corrected into "isotropy".

Reviewer p.3, below Eq7: AT VARIANCE WITH (not "conversely", which is used alone as an adverb) metallic materials.

Authors Corrected

Reviewer But I really struggle to understand the rest: "the originality is addressing the "plastic dilation" [Ognedal et al., 2014] i.e. the volumetric plastic strains." The originality of what? What has to do with metals vs. polymers? If it was done in 2014, why is it original? This is an example of a quite obscure and unsubstantiated statement. I'm sure the authors had something smart and clear in their mind, but we don't get it.

Authors The sentence was rephrased as follows in the revised manuscript: "At variance with the metallic materials where the plastic strain is uniquely related to the deviatoric parts ($\epsilon_{devT}^P, \epsilon_{devL}^P$), the specificity here deals with an account for the "plastic dilation" [Ognedal et al. 2014] i.e. the volumetric plastic strains ($\epsilon_{volT}^P, \epsilon_{volL}^P$)."

Reviewer Section 3.1: this is a first example of small things which can help the life of the reader. Please highlight the skin and core in fig 1a using some yellow dashed lines or similar, so that we know what you're referring to in the picture

Authors Following the reviewer request, figure 2a (new number) was annotated. Moreover, the paragraph explaining the skin-core effect due to the process was extended in section 3.1: "Optical microscope examination on microtomed slices of the rectangular section is illustrated in fig.2a. This latter figure shows a through thickness cut from a microtomed thin layer of the PP material at a large scale. The outer surface is indicated and a yellow dotted line has been drawn to approximately separate the skin

part from the core. This skin-core effect as detailed in [Laiarinandrasana et al., 2016b] is due to the temperature gradient during the crystallization process: the surface, in contact with the mould, experiences a higher cooling rate resulting in smaller spherulites. The transition from these small spherulites at the surface to the core is characterized by some oriented stripes, corresponding to trans-crystalline zones [Kantz et al., 1972, Assouline et al. 2001]. Here, the skin is composed of both layers of small spherulites and the trans-crystalline zone; the end of this is characterized by the yellow dotted line."

Reviewer In section 3.3 the cylindrical coordinate system z, r, θ is introduced. I really failed to understand why we need such a system, especially since all the images are treated as 2D maps on which coordinates work like a common cartesian xy reference system; they're not used as cylindrical ones. Either it's not needed (and thus unnecessary complicate), or I completely missed it.

Authors The subject being the spherulite deformation which presents transverse isotropy the cylindrical coordinates was considered to be the more relevant. The tensile direction z being the axial or longitudinal one. For the specimen with cartesian xyz reference mentioned by the reviewer, x and y should be assigned to r and θ axes, respectively.

Reviewer In the second case, I would recommend including a sketch of the 3D volume and reference set from which the images are taken, because the whole paragraph is not clear. The text description of side and top views of something which is of uncertain position and orientation is REALLY hard to follow without it.

Authors The reference system has been systematically indicated in the figures. The planes corresponding to side and top views were indicated as (r, z) and (r, θ) planes respectively. It should be noted that in fig.10 histograms, a comparison between the diameters through r and θ directions were made to ensure the circularity of the spherulite in the (r, θ) plane (top view).

Reviewer Section 3.4: all the features that the authors spend so many word to describe MUST be highlighted clearly on the relevant figures, to make sure the reader is looking at the correct ones, and also to make it much easier to follow. Also, it is not clear to me why the image analysis is split between the Material and Methods and Results sections, depending on the resolution. My suggestion is to create a single section with the sketches (see later), all the images irrespective of the resolution, commented with highlighted features.

Authors Following the reviewer instruction, the structure of the paper was modified: section 3.4 was moved to section 4.1, where the separation between the two resolutions was removed. Additionally, as mentioned above, the figures were annotated and the details of the approach were further commented in the revised section (in red in the revised manuscript but not reproduced here because these paragraphs were added in various sections and sub-sections). The sketches were added in section 2 entitled "Background", divided into: new section 2.1 (Voiding inside a spherulite) and section 2.2 (Finite strain formulations)

Reviewer How can you be sure that the voids are originated by rigid particles and not by some other kind of defects? We are given no evidence to support this conclusion. It is not that important, but unless there is evidence we shall not point out such a precise cause.

Authors Synchrotron Radiation Computed Tomography is based on the density of the matter crossed by the X-Rays. The rigid particles are bright and voids are in black. The patterns observed in fig.6 where bright "dots" surrounded by a diamond/ellipsoidal shaped black pixels (largest dimension in the tensile direction) are representing voids nucleating from the dense particle inside. The authors removed the word "rigid" in the revised manuscript.

Reviewer Figures 6 and 7: please highlight for us the boundary of the 40 spherulites! You already did it, show it to us (possibly with a more contrasting color than figure 7, like yellow which should replace red).

Also highlight the voids and the stripes. All the features that you see should be made evident to us as well, 'cause at present I'm not sure I'm able to spot all of them.

- Authors** In fig.7b the boundaries of the 4 spherulites commented in the manuscript were drawn...in red. The contrasting color being highly dependent on the initial brightness/contrasts of the initial data sets. At this higher resolution, these boundaries are better highlighted than those at lower resolution of $0.7 \mu\text{m}$. The authors hope that from these indications, the reviewer and the readers would better understand (and not forced to) how to recognize the 40 (approximate number) spherulites in fig.7a.
- Reviewer** End of section 4.1: "Clearly, the horizontal cut was done at the end of a polar fan". Another statement which is not justified. To me, it's not clear at all. I'm sure it is for you: please show it to us as well! At this stage, we would really benefit of an idealized sketch of the structures which are going to be discussed, a parallel to draw with the actual image. The authors should present their idea of the spherulitic structure complete with polar fans, stripes, voids, fibers. Once we know what they're talking about, then we can effectively recognize those structures in the images which, as the authors themselves point out more than once, are not always well-enough resolved. This "Clearly" at present really clashes with the fact that it is quite hard for the reader to reconstruct the complex structures the authors have in mind and only describe with words from the reconstructed images.
- Authors** As mentioned above, section 4.1 was strongly modified. These modifications were edited in red in the revised manuscript. Having added the Background section 2.1., the authors hope that the readability of the approach was improved.
- Reviewer** The use of different scales for the different sub-images abc of figures 9-10 contrasts with the intent of the authors to show differences in dimensions as we move towards the necking region. Better to use the same scale for all 9s and another one for all 10s, so that the difference is immediately evident.
- Authors** Figs. 9-10 became 10-11 in the revised document. Actually, the evolution of dimensions through the z direction towards the necked region is shown in fig.12. To plot this latter, a "statistical" parameter (mean, median, mode) had to be selected from the histograms. To better show the relevance of the selected parameter (here, the mean value), the classes (intervals) for the abscissa of the histogram have to be well optimized.
- Reviewer** If the authors want to draw a comparison between these data and classical measurements such as DIC or extensometry, we need to see those measurements. Otherwise it's another unsupported claim.
- Authors** The authors would remind that: i) the scope of the work is focused on the average spherulite within the core layer; ii) the data sets are in 3D; iii) and the measurement at the surface is carried out on the skin layers. The skin VOI is assumed here to reflect what can be measured at the plane at mid-width of the sample in terms of longitudinal and transverse displacements. The warping of the outer surfaces of the specimen would suggest that neither extensometry nor DIC technique can reach the precision obtained here.
- Reviewer** Fig. 11: was this done for a single measurement? Can't it be repeated for several spherulites to show data dispersion, to demonstrate if differences are significant?
- Authors** (Fig.11) Now Fig.12: the number of spherulites participating in the averaging operation is specified in the histograms (fig. 10 and 11). This is also the reason why these histograms were not at the same scale. The deterministic approach (Continuum Mechanics) based on the average spherulitic RVE should be related to these data. Of course, a stochastic approach can be developed but it is out of the scope of this work.
- Reviewer** p.8, L2: the "originality" of the present work (which I think should be instead its "novelty") is to verify through-thickness homogeneity, but to me it's not clear if we are really doing it. Since by construction the displacements of skin and core are similar, what are we verifying? I'm not sure I understood correctly what the authors mean.

- Authors** The term "originality" was changed into "novelty" as required by the reviewer. The longitudinal displacements of skin and core are similar, but the transverse ones are very different. For the average spherulite, both displacements are different from the skin and core material. So, the transverse displacements (related to the volume change) showed heterogeneity.
- Reviewer** Also, later on below equation 18, I think it is misleading to write about "measured" elongations since as far as I understand, they are calculated (although separately for the spherulite). This whole last part is really not very clear to me, maybe the authors can try to explain what they're doing in a more effective way.
- Authors** The elongations consisted of the measured lengths (Table 2) divided by the gauge lengths (Table 1) for each considered VOI. The definition is just (engineering strain + 1). However, the term "measured" was removed in the revised document.
- Reviewer** p.9: a question to the authors regarding the last results. Are there any techniques (e.g. optical, or spectroscopic) which could confirm at least qualitatively the volume variations estimated from the analysis? Not that this should be included in this work, but I think it would be interesting to suggest possible avenues for cross-checking the results.
- Authors** Comparisons with other experimental techniques like picnometry, video-traction, incoherent polarized steady-light transport have been done by the authors on other studies. Most of them assume the homogeneity of the measurement through the volume of the tested sample. To the authors' knowledge, only experimental techniques allowing local measurements like 3D imaging techniques (Tomography, X-Ray diffraction) enable to analyze the heterogeneous volume change through the VOI.
- Reviewer** End of section 5.1: "much more smaller spherulites can deform by rolling of the spherulites each other provoking a "flow" of the overall material". No idea of what you are describing here. Please support with some sketches, otherwise it is really hard for us to visualize what you are writing about.
- Authors** This sentence has been modified as follows: "The skin, composed of much smaller spherulites can deform by the rolling/slipping of these spherulites each other. The "flow" of the skin layer can then be attributed essentially to shear without voiding phenomenon. Therefore, the skin shrinks less than the core with coarse spherulites".
- Reviewer** Since I got a bit lost in the previous part, I struggled a lot to follow the consequences on true strain and true stress. If there is a logical connection between the two parts, it needs to be better explained.
- Authors** So far, the elongations (engineering strains) were discussed, showing triaxial aspects. The large strain formulation in continuum mechanics approach works on the true stress/strain tensors. The aim of the section is to extract the true plastic strain tensor, including the volumetric one (Equation. 21). A simple Von Mises plasticity model is not sufficient to obtain the true stress accounting for this plastic volumetric strain. Other class of model is necessary.
- Reviewer** AI analysis: it seems to me what you are suggesting is simply an automated tool to analyze images. This is not AI or machine learning, which would capitalize on that data - possibly over a much larger dataset - to extract correlations between physical variables not driven by the current analysis but from evidences in the data themselves. I think the reference to AI is misleading (or you haven't explained to us what you want to do with the data). I think section 5.3 should be removed, except for saying that an automated tool would greatly speed up the analysis, possibly making it more objective (if suitable criteria/thresholds can be identified, which is not trivial)
- Authors** The title of the section has been modified: "machine" learning instead of "deep" learning. The present work followed two steps. The first one aimed at the identification/recognition of the boundaries of the spherulites using slices (side/top views) from the whole data set. The second step consisted of using these patterns to recognise the largest amount of spherulites in the data sets at lower resolution.

It seems that the same steps are followed by a machine learning process. For instance, the first step here would be used to provide to the machine learning technique a catalog of the patterns (drawn in red in figs.8c and 9c) so as to initiate the learning process. Then, in the second step, the AI should then be able to recognize by itself all the patterns included in the whole data set. This technique should even be able to deduce from 2D data here the boundaries of spherulite in 3D, which would require another step of "learning" process. This approach can be called AI for the segmentation of tomographic 3D images. This is the authors' point of view. The section was then maintained in the revised manuscript, but considered as a future work. But if the reviewer finds it useless or misleading, the authors would agree to remove the subsection before the final submission.

Reviewer As for the minor language/editing improvements, a few examples of points to address: Figs.11-12 Please use a consistent symbol/colour code for the different datasets (skin, core, spherulites)

Authors Done one figs. 12-13, extended to fig.14.

Reviewer p.1, L6: A 3D imaging technique, or techniqueS

Authors Done

Reviewer p.1, L9: what is a "smooth - therefore uniaxial" specimen? What do you mean?

Authors "Smooth" specimen was mentioned here by opposition to Necked/Notched specimen, once it experiences necking. In the revised version it was modified as : an "uniaxial specimen".

Reviewer p.1, L18: resultS

Authors Done

Reviewer below Eq1: lambda suffix should be theta in symbol, in accordance with the rest of the paper

Authors Done

Reviewer p.3, L1: for AN incompressible material

Authors Done

Reviewer p.3, last paragraph: "the same transports as" what does that mean?

Authors "Transport" is the term introduced in finite strain formulation (Deformation gradient transport) to relate, for instance, the Cauchy stress to the Piola Kirschhoff stress. For the sake of clarity, in the revised text "transports" was changed into "operations".

Reviewer section 3.2, second paragraph: regardless OF the distribution

Authors Done

Reviewer section 3.4, L2: "vollate one deformed spherulite"? What does that mean?

Authors Apologize: this a typo due to awkward changes of word "iso" into "vol". The right word is "isolate" (modified in the revised manuscript). The same mistake on the term "voltropy" instead of "isotropy"

Reviewer later on, "the remainder being hidden by the "matter" let by the clip box": not clear, what does that mean?

Authors The sentences were rephrased in the whole paragraph which was modified (text in red)

Reviewer next line: the top view REVEALS (and not "let appear")

Authors Done

Reviewer section 4.1: the tensile direction IS vertical

Authors Done

- Reviewer** second paragraph: rearrange sentence with "can be observed" at the end, where it should be; voids and matter (with no final "S"); diameter increaseS. This is an example of a sentence with minor errors but the way it's written it takes 2 or 3 times to figure out what it means.
- Authors** The sentences were modified in the whole paragraph which was modified (text in red)
- Reviewer** Last paragraph: three voids nucleated (remove "having")
- Authors** Done
- Reviewer** lamellae, not lamMellae (all instances); among them there's also a labBelled
- Authors** Done
- Reviewer** Gauge lengths at three scales: the following sentence is unintelligible. Also add a "therefore" before next "only two elongations"
- Authors** The sentences were modified as follows: "The elongation λ is defined as L/L_0 where L_0 is the gauge length of the RVE. Table 1 summarizes this gauge length for each VOI. These values will be used in the following"
- Reviewer** p.8, beginning of fourth paragraph: the ratio between the MACROSCOPIC elongation and that at the scale of the spherulite...
- Authors** Done
- Reviewer** p.9, end of second paragraph: when DIC HAS BECOME OF COMMON USAGE
- Authors** Done
- Reviewer** Results are many, the Discussion is in my opinion only one (no "S")
- Authors** Done
- Reviewer** In the Discussion, last sub-bullet point: AND the evolution of the height
- Authors** Done
- Reviewer** Next page, first bullet point: matter left (not left matter)
- Authors** Done
- Reviewer** In the references, all titles have polymer names in small caps (hdpe, pvc...)
- Authors** Done

Review of version 2

Permalink: [hal-04100630v2](https://doi.org/10.1006/30v2)

Reviewer 1 (Luca ANDENA)

- Reviewer** I want to congratulate the authors who did a wonderful job to revise the paper and answer all of the (many) points raised by the first review. Since their results should be of great interest to the scientific community, I thought that they deserved an equally good presentation. I hope they agree that the significant extra effort required from their side contributed to an actual improvement of the revised version! The paper in its present form will make a fine addition to JTCAM.

Reviewer 2 (Anonymous)

- Reviewer** The paper is focused on the analysis of the results obtained from Synchrotron Radiation Computed Tomography carried out on the necked region of an isotactic polypropylene specimen stretched in uniaxial tensile loading conditions. The morphology of deformed spherulites was evaluated

and by measuring the longitudinal and transverse elongations of the spherulites in the necked region, an expansion of the material was initially observed followed by a subsequent compaction. The mechanisms responsible of this change in volume evolution have been proposed. Further, a constitutive model considering the volume change dependence on the macroscopic applied strain was proposed.

The work presents interesting and original results and deserves to be published, but a minor revision is necessary, especially aimed at the introductory part.

The article is in fact a continuation of work reported in a previous article of which some of the authors of this paper are authors. But, although this aspect could be of help to the reader to frame the work, this is not clearly written in the article. It is difficult to understand (i) that this is the analysis of a single sample (as I think it is) among the many studied in the previous work, (ii) how the material tested in traction was prepared, how the sample was prepared and how the sample observed by SRCT was prepared, (iii) how Tomographic analysis was carried out, (iv) what was observed in the previous work, the starting point of the new work.

Aspects related to points (i, ii, and iii) should be added in section 3.1.

Aspects related to point (iv) should be added in section 2.1. For similar reasons, the comments made by the authors in the first 22 lines of section 5.1 could be moved to section 2.1.

The title should also be reworded: the work focuses on isotactic polypropylene and should not be generalized to all semi-crystalline polymers.

There are some English or typing errors: a careful re-reading of the text is suggested.

Authors All requested minor revisions have been made in the last version of the paper.

Editor’s assessment (Anna PANDOLFI)

The paper discusses Synchrotron Radiation Computed Tomography results on an isotactic polypropylene specimen stretched in uniaxial tensile loading conditions. The exam of the results allowed to detect the mechanisms responsible of the volume evolution of the specimen under loading and to propose an innovative constitutive model. The two reviews were accurate and deep and the authors provided an excellent job in revising the paper by addressing all the points raised by the reviewers, and improved the overall presentation of the research done.

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