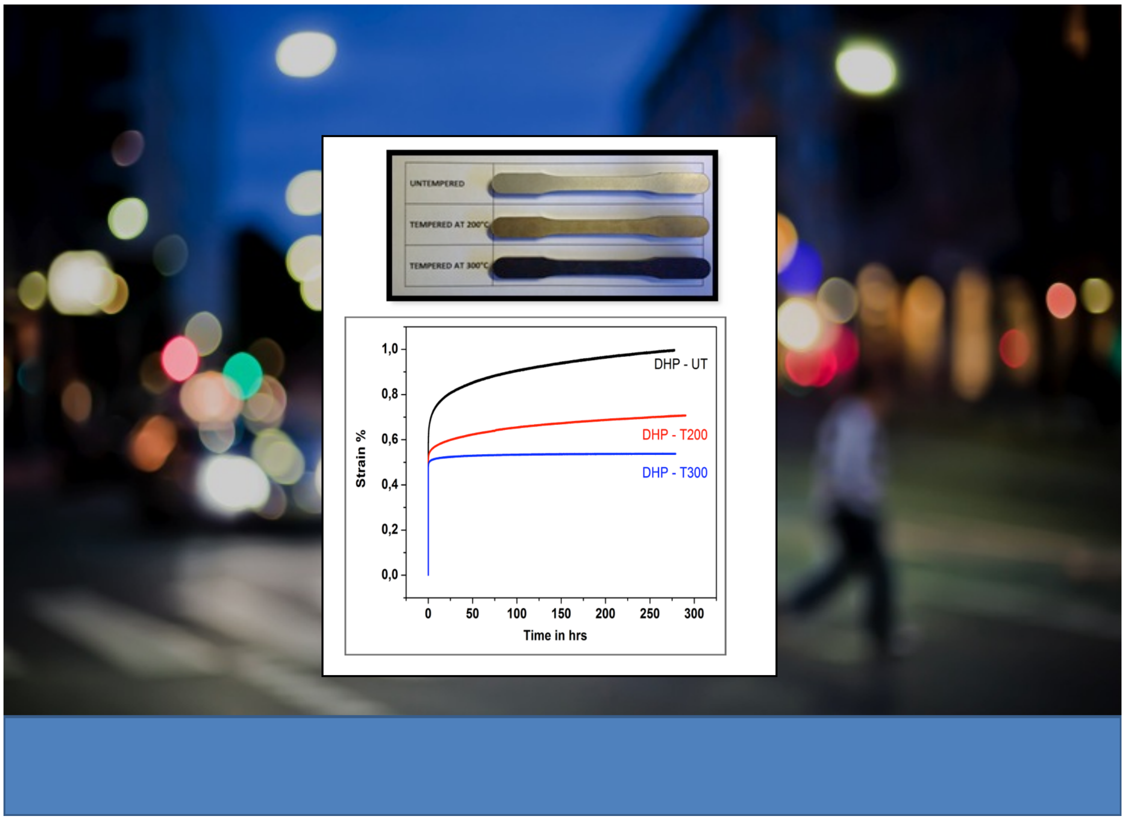
Sintered Part with High Static Loading Capacity – Effect of Tempering on Deformation Stability at Application Conditions



Project within Sustainable Production

Author: Lars Nyborg, Chalmers University of Technology, Materials and Manufacturing Technology

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FFI

Content

FFI in short

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1. Executive summary

The development with fewer engine families for heavy trucks is an important driving force for increased use of sintered parts, owing to large series cost benefits. Applications are those primarily involving demands on static load/bearing capacity. Today, sinter parts with their good tolerances can be realized with mechanical strength that well meets the required product demands for such applications. In this context, it has been confirmed that tailoring of the tempering is a functioning means of making PM steel sustainable towards high static loading in potential automotive applications. The project has also developed further theoretical understanding behind the possible mechanism that could be responsible for the phenomena of static loading sensitivity with respect to creep/relaxation resistance at only slightly elevated temperature. The results have been disseminated to a broader audience within Volvo and Höganäs with great attention and selected results have also been communicated at EuroPM2013. There is now need for further implementation oriented research to explore how materials selection could be further improved, how to settle a further generic understanding of the addressed issue and how to expand the use of PM parts for heavy truck applications. The groups involved in the project from these companies have expressed their clear interest in finding ways of realizing a regular FFI-project and establishing a long-term development based on the findings of the project. Furthermore, the issue addressed in this project is to large extent not publically known or considered within the PM community in at least Europe. Hence, the R&D done within this hypothesis project can be viewed as a unique contribution to the further development of PM applications.

2. Background

Technological and Industrial Context

The development with fewer engine families for heavy trucks is an important driving force for increased use of sintered parts, owing to large series cost bernefits. Applications are those primarily involving demands on static load/bearing capacity. Today, sinter parts with their good tolerances can be realized with mechanical strength that well meets the required product demands for such applications.

The PM technology is an attractive means of cost efficient realization of different automotive components. The PM technology via compaction and sintering of metal powder is also one of the most efficient ways of component manufacture with respect to energy efficiency and raw materials utilization; the energy demand has been estimated at 30 MJ/kg and the raw materials utilization is 95%, see Fig. 1. In this way PM technology is higly competitive compared to casting, forging, machining, etc.

Sintered parts are manufactured by powder compaction and subsequent sintering of the compact at high temperatures (usually 1120°C for maximum about 30 min). Product shapes and dimensional tolerances are largely set during the compaction, while the sintering creates the necessary metallic bonding between the metal particles. To maintain the shape and the inherent excellent dimensional tolerances, there is usually very little if any shrinkage during the sintering. This means that the sintered part will have certain porosity, usually about 10% which means final density of 7.0-7.1 g/cm^3. With different techniques (e.g. warm compaction, high temperature sintering, materials design, etc), the final density can be raised to 7.2-7,4, but at the expense of increased cost. The PM technology of today can deliver appropriate solutions with respect to nominal material properties, which meet the product requirements for large number of automotive applications. One such kind of application is when the main demand is static loading capacity and fatigue resistance is not necessarily of prime concern. The inherent porosity can also be an advantage as it means potential weight savings, provided that material performance and product design are optimised. Manufacture of sintered parts for high static loading application include hardening and tempering.

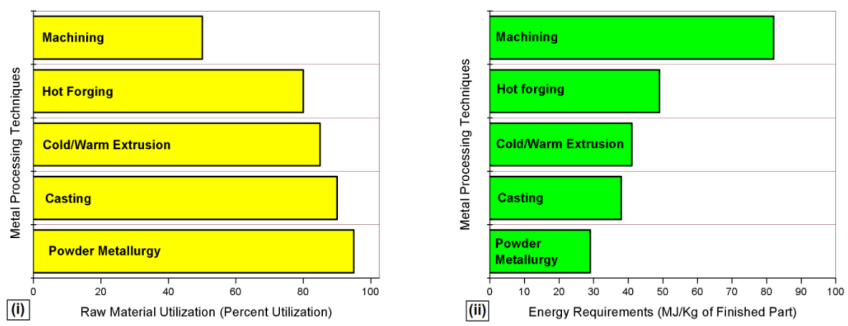

Fig. 1. Environmental performance characteristics for sintered parts manufacture compared to other kinds of processes (data using Cambridge Engineering Selector).

It has, however, been found by both Volvo and Höganäs that PM components can show unexpected relaxation/deformation at static loading and only slightly elevated temperature (T<120°C). The reasons for the phenomena are largely unknown, but it can be understood that the stability of the microstructure in hardened and tempered condition plays an important part. The importance of the microstructure stability has been further underlined by prior preliminary experiments (Volvo, Höganäs), which indicate the modified tempering could be a feasible solution.

Scientific Issues

The basic issue as said above is that the microstructure of sintered steel in hardened and tempered condition is essential for its capacity in sustaining static loading without long-term deformation owing to structural changes, relaxation or creep.

The microstructure in question is usually slightly temperared martensite with high hardness and certain toughness. In principle there is primarily martensite, but certain amount of so called retained austenite can be present since sintered steel usually has carbon levels of at least 0.5 weight%. The retained austenite can when being deformed be transformed to martensite. Locations with reatined austenite in the material are however surrounded by the martensite formed during hardening and the volume expansion at its formation means that the retained austenite is stabilised. Whether a sintered steel with its inherent porosity would show any special behaviour has not been found from literature. Deformation induced transformation of austenite is as said associated with local volume expansion. To what extenet presence of retained austenite is an important factor for the structural stability/relaxation resistance/creep resistance is if interest to clarify.

Tempering of sintered steel is commonly done at temperatures of between 100 and 200°C. Preliminary observations have indicated that the modified tempering, e.g. higher tempering temperature, may have a retarding effect on the observed deformation phenomonenon. With increasing tempering temperature, more carbides are formed, while the carbon level of the martensite is lowered as well as any retained austenite is also affected. Potential impact of such effects or other changes are also of importance to understand better including how they may correlate with the potential relaxation/creep.

Finally, we have the creep deformation in classical way. Creep is recognized by persistent deformation through diffusion and deformation (dislocation movement) processes. Creep can be divided into so-called primary, secondary and tertiary creep. Creep fracture requires activation of tertiary creep, so creep at only slightly elevated temperature can thus only result in deformation where the actual load level is crucial. It could be viewed unlikely that there sohould be noticeable creep in steel (in hardened and tempered condition) at only slightly elevated temperature (<120°C) for those times and loads of relevance for applications and experiments considered here. However, it should be noted that limited low temperature primary creep has been observed for conventional steel (previous diploma thesis work at Chalmers) and it can also be pointed out that creep rate is initially always fast until a minimum is reached whereafter secondary creep takes place. This means that initial creep processes may be active and may lead to certain deformation depending on load level at only slightly elevated temperatures. The fact that sintered steel owing to its porous structure may mean local stress concentrations could also mean that creep could be locally stimulated. To what extent initial creep can be involved for sintered steel parts of concern for static load bearing capacity in automotive application needs to be investigated.

3. Objective

Sintered steel parts are today higly attractive for a number of components for heavy truck applications considering the development with fewer engine families and thus larger series. Of particular interest is the long-term load bearing capacity with respect to relaxation of the sintered steel in the intended application. The hypothesis is that the optimisation of microstructure of hardened and tempered sintered steel must be better and further developed. Included here is to clarify whether a number of mechanisms as otlined above can be active or contribute. One goal is to establish the rrole of the tempered microstructure itself and another goal is to show to what extent creep processes can contribute to the lacking deformation stability observed (at temperatures of below 120°C).

The project is of prime importance for wider use of sintered parts in e.g. heavy trucks. Answering the hypothesis including possible identification of mechanisms behind the mentioned static load deformation sensitivity is a necessary for future development of PM-based manufacturing and product concepts for heavy vehicles.

It should emphasized that this project originates from phenomena observed in practical tests. At static loading (below the load necessary for plastic deformation) dimensional change has been noticed. Industrial development work has addressed the issue, but convincing explanations are still lacking. In dialogue with Chalmers the current project has then been developed as a first step towards further implementation of PM sintered steels.

4. Project realization

Project partners have been Chalmers (Materials and Manufacturing Technology), Volvo and Höganäs and the project duration has been between 2012-10-01 to 2013-09-30. The strategy has been as follows:

- The partners have planned the work by selecting test materials, agreeing on main R&D questions in more detail, dividing of work and responsibilities
- A MSc thesis has been initiatied and connected to the project with work partly performed at Chalmers and Volvo
- Chalmers has developed methodology to creep test sintered test at slightly elevated temperature
- Chalmers has developed a research plan involving the running of creep tests, materials characterisation and theoretical analysis
- Höganäs has manufactured test specimens and performed mechanical testing in support for the project goals realization
- Volvo has complemented the work at Chalmers by own tests using a specially designed test set up applied on component-related test piece.

The distribution of work can be seen from Fig. 2. below.

Fig. 2. Distribution of work.

Höganäs	**Volvo**	**Chalmers**
Tensile sample preparation and testing	Creep testing on test parts	Creep testing on tensile specimens & characterisation

5. Results and deliverables

5.1 Delivery to FFI-goals

This hypothesis project addresses Milestone 2 (2020): *Light materials and new processes: large series manufacture of components for different kinds of vehicles and other alternative drivelines through new manufacturing processes*, but also the Milestone 3 (2025): *Environmentally neutral production, optimised processes*. The basis is that PM as net-shaping technology involving powder compaction and sintering (as outlined in the 'Background' section) is among the most energy efficient ways with highest raw materials utilisation for component manufacture. The further development of usage of PM would also mean potential cost benefits for large series components in heavy trucks.

5.2 Technical and Scientific Results

Test Matrix
It was decided to concentrate on so-called diffusion-alloyed powder grade, namely Distaloy HP (4 wt% Ni, 2wt% Cu and 1.5 wt% Mo) which was prepared with 0.6 wt% graphite, compacted and sintered into test bars by Höganäs involving so-called sinter hardening. Out of these samples, following test variants were then created (Table 1).

Table 1: Test variants included in the creep testing matrix at Chalmers

Samples	Distaloy HP	Denominations	Description
1	DHP	UT	Untempered
2	DHP	T200	Tempered at 200°C for 1 hour
3	DHP	T300	Tempered at 300°C for 1 hour
4	DHP	DC-UT	Deepcooled in Liquid N_2 and untempered
5	DHP	DC-T200	Deepcooled in Liquid N_2 and Tempered at 200°C for 1 hour
6	DHP	DC-T300	Deepcooled in Liquid N_2 and Tempered at 300°C for 1 hour

Design of experiments
The idea (based on relevance criteria) was to perform "creep testing" at below 100-150°C at maximum about 90% of the yield stress. Tensile tests were therefore first performed at Höganäs to find out the static mechanical properties of the different variants (Fig. 3). The creep testing was then accordingly done with 20 kN, which corresponded to 622 MPa or at least 90% of the yiled stress for the least strong variant.

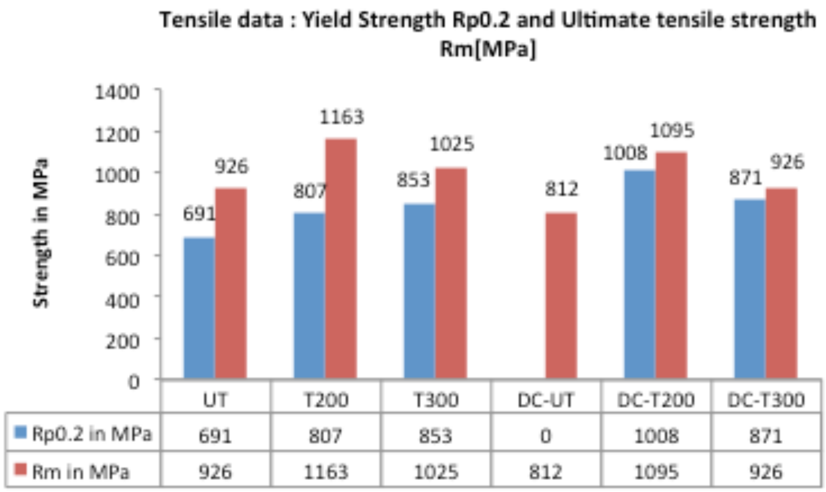

Fig. 3. Tensile test data for variants of tempering investigated.

The test and evaluation matrix for the experiments at Chalmers is shown in Table 2. Here, only a selection of the most important results and observations will be reviewed.

Table 2. Test and evaluation matrix for experiments at Chalmers

Material : Distaloy HP		DHP			DHP		
		UT	T200	T300	DC-UT	DC-T200	DC-T300
Metallography	Before Creep	✓	✓	✓	✓	✓	✓
	After Creep	✓	✓	✓	-	-	-
Fractography - SEM		✓	✓	✓	✓	✓	✓
Hardness	Microhardness	✓	✓	✓	✓	✓	✓
	Apparent Hardness	✓	✓	✓	✓	✓	✓
XRD-Austenite analysis		✓	✓	✓	✓	✓	✓
Tensile testing		✓	✓	✓	✓	✓	✓
Creep testing	Cyclic creep	✓	✓	✓	*	✓	✓
	3.6×10^5 seconds (100 hours)	✓	✓	✓	*	✓	✓
	1 million seconds (~ 277 hours)	✓	✓	✓	*	-	-

A test rig was designed and constructed for a universal mechanical testing machine equioment with a temperature control chamber. This allowed long term creep testing at only slightly elevated temperature. Such tests were performed for up to approx. 300h. Results were then also compared with component-like tests at Volvo.

Results and Discussion

Characteristics of test variants

Figure 4 below compares the hardness data for variants before and after temperings as well as after creep for 1 million sec. at 120°C. T It should be pointed out first that the sintered material is so-called heterogenous material with Ni-rich areas (austenite) besides tha main constitutents of martensite and bainite, see e.g. Fig. 4. The most important observation is then that tempering at higher temperature appears to give a more stable microstructure, which is not affected in terms of hardness during the low temperature creep testing.

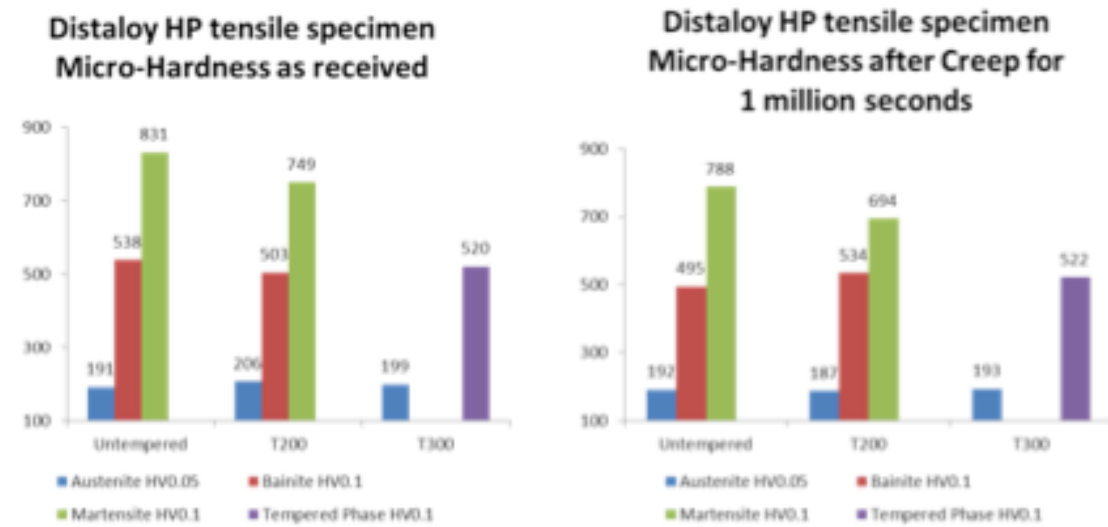

Fig. 4. Hardness data before and after temperings as well as after subsequent creep testing at 120°C for 1 million sec. at 20 kN load.

Now, it can be anticipated that the tempering at higher temperature also has a beneficial effect on the plastic strain resistance and indeed this is also the case as shown in Fig. 5. This figure compares the results for un-tempered and with two variants of temperings. Clearly, tempering at 300°C means that the secondary creep is basically eliminated (part after the initial strain increase).

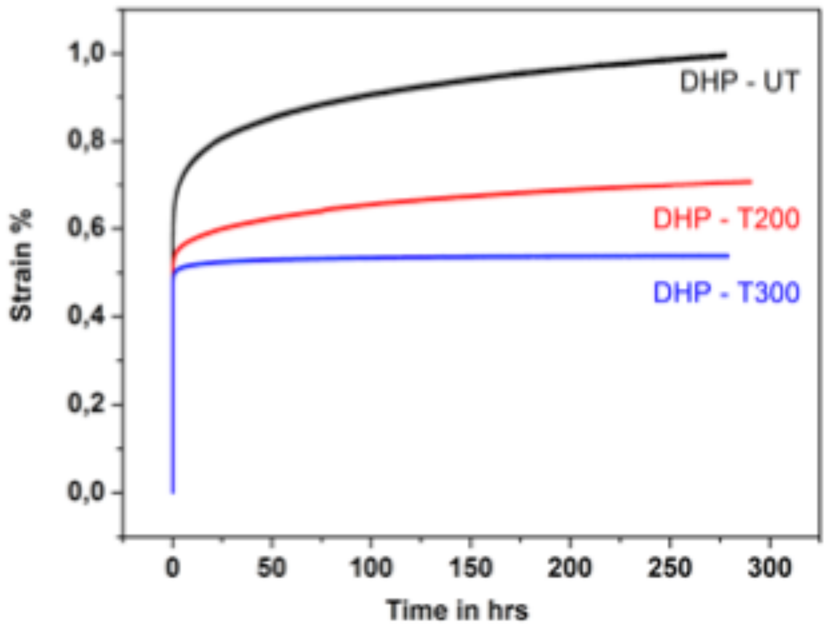

Fig. 5. Creep tests of different variants of sinter-hardened Distaloy HP samples.

Tests were also performed to clarify whether the observed creep data would correlate with dimensional changes measured on test specimens. A special procedure was deleloped for this involving the assessment of distances between indentation marks. The results of the latter are shown in Fig. 6. Quite good correlation with measured creep data is obtained, hence indicating the appropriateness of the applied approach. Figure 7 below illustrates the assesment of plastic strain from positions of identations marks on a sample.

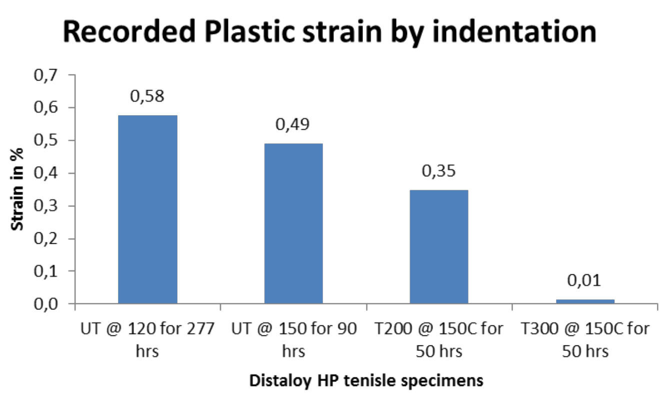

Fig. 6. Plastic strain for creep testing obtained via assesment of distances between indentation marks before and after testing.

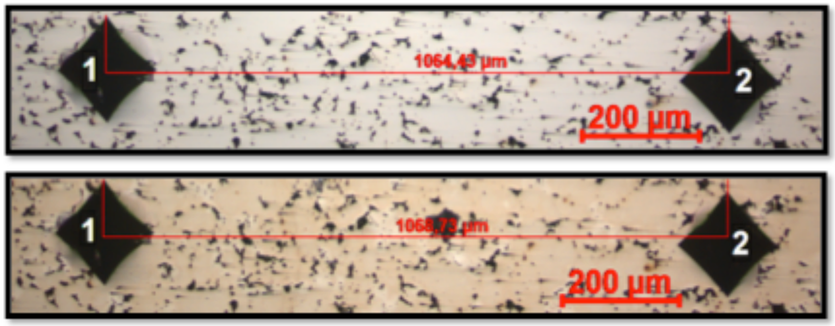

Fig. 7. Illustration of measurement of plastic strain from comparison of indentations marks on a test specimen before and after creep testing at low temperature (un-tempered condition tested at 150°C for 277 h).

One important issue is to see whether existance or change in presence of retained austenite could be of importance. Figure 8 below summarises the results of the austenite content assessments by means of X-ray diffraction analysis of the different variants. As can bee seen the tempering at higher temperatures lowers the total content of austenite. However, the austenite content before and after creep testing was not different for a specific variant (not shown). It should also be noted that the austenite content measured may represent the sum of Ni-rich austenite and retained austenite. For this reason deep cooled samples were also investigated. Such samples should not contain the latter kind of austenite, whereby much lower austenite content is indicated, see Fig. 8.

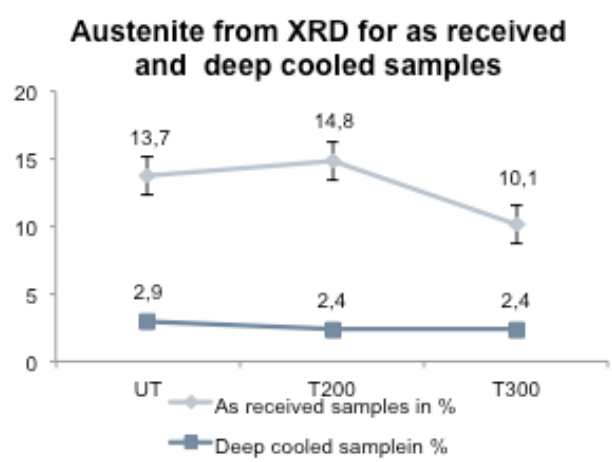

Fig. 8. Assessment of austenite content before and after different temperings for the Distaloy HP steel.

For comparision, test results from Volvo are also illustrated in Fig. 9. These more ”component-like” tests in a special laboratory set-up confirms the results from Chalmers presented above.

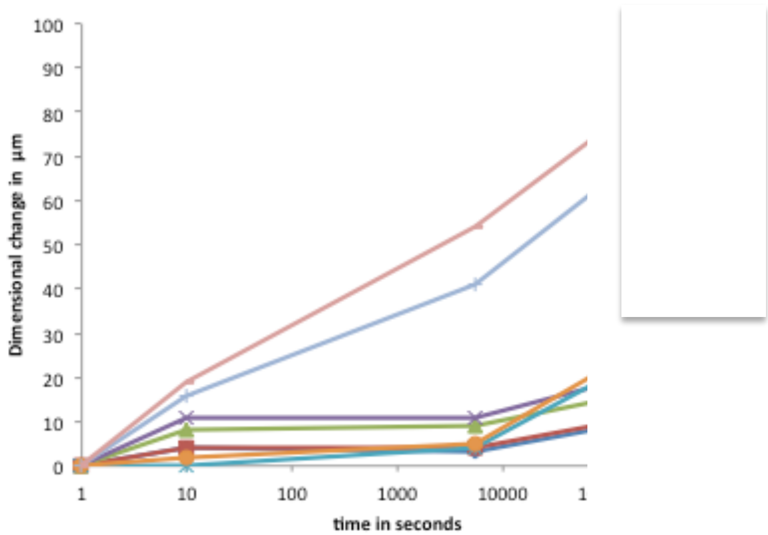

Fig. 9. Dimensional change tests of more ”component-like” sintered specimens using a special set-up. Same material as for Chalmers tests.

6. Dissemination and publications

6.1 Knowledge and results dissemination

The project has confirmed that tailoring of the tempering is a functioning means of making PM steel sustainable towards high static loading in potential automotive applications. The project has also developed further theoretical understanding behind the possible mechanism that could be responsible for the phenomena of static loading sensitivity with respect to creep/relaxation resistance at only slightly elevated temperature. The results have been disseminated to a broader audience within Volvo and Höganäs with great attention and selected results have also been communicated at EuroPM2013. There is now need for further implementation oriented research to explore how materials selection could be further improved, how to settle a further generic understanding of the addressed issue and how to expand the use of PM parts for heavy truck applications. The groups involved in the project from these companies have expressed their clear interest in finding ways of realizing a regular FFI-project and establishing a long-term development based on the findings of the project. Furthermore, the issue addressed in this project is to large extent not publically known or considered within the PM community in at least Europe. Hence, the R&D done within this hypothesis project can be viewed as a unique contribution to the further development of PM applications. As already mentioned in the “Background” section, the development towards fewer engine families means that the number of same components increases. Then PM products that are highly competitive from total cost point of view for larger series become increasingly interesting. Still, the PM technique itself allows for flexible production and design and hence a user as Volvo would benefit from such conditions. Adding parts manufacturers/suppliers to the existing constellation would be beneficial for possible next phase of efforts. This could also be a way to further strengthen existing PM parts producers in Sweden as these are few and not necessarily competitive in offering their products to the OEMs. The PM parts manufacturing field in Sweden can be viewed as a "Gap problem". Sweden is the dominant actor in the world when it concerns powder fabrication with about 25% of the world production. It is also basically the dominant actor in the area of heavy vehicles where PM parts are used and can be used even more in future. Hence, there should be significant potential in filling the gap in between by PM parts manufacturers provided they have the necessary capabilities and manufacturing excellence.

6.2 Publications

1. K.B. Surredi, M.V. Sudaram, E. Hryha, H. Karlsson, M. Andersson, L. Nyborg, "Low Temperature Creep Behaviour of PM Components under Static Loading Conditions", Proc. Of EuroPM2013 (Gothenburg Sep 15-18), EPMA, Shrewsbury.

2. M.V. Sudaram, "Low Temperature Creep/Relaxation Behaviour of PM Steels under Static Load" , MSc Thesis, Chalmers University of technology, 2013.

7. Conclusions and future research

Based on the experiments and analyses peformed, the hypothesis has been clearly confirmed and we have settled a basis for potential further development of PM application potential for heavy truck components. The major conclusions are as follows:

- Creep/relaxation was observed under simulated conditions
- Sinterhardened samples without tempering exhibits more plastic strain than tempered samples
- Large plastic strain in untempered samples is due to the unstable microstructure
- Samples tempered at 300°C shows negligible amount of plastic strain due to stable microstructure – hence tailored tempering is practical way of solving the creep sensitivy issue
- Sinterhardened - Deep cooled + tempered samples shows stable and negligible plastic strain during creep → might be due to retained austenite transformation
- Tests on "component-like" test pieces → similar behaviour → untempered samples exhibits higher plastic strain

Based on these results, we have identified following important issues for future:

- Creep tesing for another sinterred steel for comparative purposes

- More precise assessment of austenite content
- Perform tests on fully densified parts by HIPping – depicting role of porosity
- More detailed study of transformation of martensite and retained austenite upon tempering
- Development of model to predict low temperature creep behaviour of PM steels

8. Participating parties and contact person

Chalmers University of Technology (Department of Materials and Manufacturing Technology)
Professor Lars Nyborg (project leader), lars.nyborg@chalmers.se
Associate professor Eduard Hryha
Dr Kumar Babu Surredi (post-doc)
Mr. Maheswaran Vattur Sundaram (MSc student)
Volvo Trucks Operations, Materials Technology
Dr. Henrik Karlsson
Höganäs AB. Global Development
Dr. Michael Andersson