18th IFHTSE CONGRESS
International Federation for Heat Treatment and Surface Engineering
IFHTSE
ANAIS
PROCEEDINGS
ISSN 1516-392X

HEAT TREATMENT OF MICRO-COMPONENTS IN A DROP-DOWN TUBE FURNACE[1]

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Abstract

To face the increasing demands in the production of micro-components concerning dimension, quantity, costs and quality new production processes must be developed. At the University of Bremen the Collaborative Research Center 747 “Micro Cold Forming” funded by the German Research Foundation was founded to deal with the resulting challenges. An elementary process during manufacturing is the heat treatment to generate desired properties of the final product, e.g. mechanical strength, for later applications. For hardening micro components with a wall thickness of 50 to 100 µm made of steel a new facility for carrying out the heat treatment during falling through a heated tube was designed to avoid problems of loading the sensitive components. In a 5.5 m high drop-tube furnace, at temperatures up to 1300°C, micro components made of sheet carbon steel can be hardened in the furnace. After set-up of the furnace and measuring the temperature distribution in the tube and the drop times, hardening tests have been performed and the microstructure of the specimens has been evaluated. For tube furnace temperatures above 1100°C, micro components made of steel quality C100 could be austenitised in approximately 3.5 seconds and be hardened by quenching in water or ambient air. The resulting microstructure is very homogeneous. No influence of the quenching method on the microstructure could be detected. An increase in Martens hardness from 2196 HM 0.05/5.0/1.0 [N/mm^2] up to nearly 6200 HM 0.05/5.0/1.0 [N/mm^2] was realized. Furthermore the hardness distribution was measured across the sample thickness.

Keywords: Drop-down tube furnace; C100; AISI 1095; Hardening; CRC 747 “Micro cold forming”.

TRATAMENTO TÉRMICO DE MICRO-COMPONENTES EM FORNO TUBULAR TIPO “DROP-DOWN”

Para atender a crescente demanda na produção de micro-componentes com dimensões precisas, alta quantidade, baixo custo e alta qualidade torna-se necessário o desenvolvimento de novos processos. Para lidar co os desafios resultantes disto, a Universidade de Bremen em collaboração com a Fundação de Pesquisas Alemã fundaram um centro de pesquisas “Micro Cold Forming” (SFB 747). Um processo elementar durante a produção para alcançar as propriedades desejadas do produto final é o tratamento térmico. Para endurecimento de micro-componentes de aço com paredes de espessura de 50 µm a 100 µm um forno novo foi desenvolvida, o qual evita problemas com a manipulação destes micro-componentes. Chapas de aço carbono podem ser térmicamente tratadas em um forno tubular com altura de 5.5 m a temperaturas acima de 1300°C. Apos ajuste dos parâmetros de forno e medição da distribução de temperatura no tubo e do tempo de queda da peça no forno, testes do endurecimento foram realizados e a microestrutura das amostras foi analisada. Micro-components de aço AISI 1095 podem ser austenitizadas acima de 1100 C durante 3.5 segundos e endurecidos por têmpera em água e ao ar. A microestrutura resultante é homogênea e não foi detectada influência de método de têmpera. Observou-se um aumento na dureza Martens de 2196 HM 0.05/5.0/1.0 [N/mm^2] ate cerca de 6200 HM 0.05/5.0/1.0 [N/mm^2].

Keywords: Forno tubular tipo “drop-down”; C100; AISI 1095; Endurecimento; SFB 747 “Micro cold forming”.

[1] *Technical contribution to the 18th IFHTSE Congress - International Federation for Heat Treatment and Surface Engineering, 2010 July 26-30th, Rio de Janeiro, RJ, Brazil.*

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1 INTRODUCTION

Development trends for micro components, such as connectors in mobile phones or fuel injectors, require a steady decrease in size, weight and production cost per part and an increase in functionality. Additionally they have to be produced in shorter cycle times with a higher output regarding quantity. At the same time a precise and reliable function over the entire product life is required, which implies a constant or even improved quality. To fulfil all these requirements new production technologies, including a perfect adaption of materials and processes, are necessary because a direct downscaling of conventional processes into the micro scale is rarely possible. The most relevant difference is a significant higher impact of the local microstructure on the material and processing behaviour due to the very low ratio between for example grain size and material thickness in the range of some ten microns. To deal with the challenges of downscaling the production processes the German Research Foundation DFG initiated the Collaborative Research Centre (CRC) 747 “Micro Cold Forming - Processes, Characterisation, Optimisation” at the University of Bremen in 2007. Overall 40 scientists from 8 different institutes participate in the interdisciplinary research in 14 different sub projects.

One important process step in the production process is the heat treatment. The heat treatment process is responsible for the adjustment of the material properties on the one hand to ensure formability and on the other hand to obtain the required mechanical properties for later application. By for example recrystallisation annealing it is possible to achieve material properties, which enable an accurate and energy efficient cold forming process. In this context it is necessary to generate a homogeneous micro structure as far as possible regarding grain size and alloying element distribution. Particularly very thin components with wall thicknesses below 100 µm are very sensitive against inhomogeneities, because the number of grains in the cross section is usually very low. A higher portion of coarse grains leads to a lower number of activatable gliding planes at external load. Hence, an inhomogeneous and coarse grain size as well as other imperfections like defects or inclusions have a much greater impact on the failure behaviour of micro components the thinner the wall thickness is.[1,2] The strain hardening by cold forming can lead to considerable material strength. However, in general a defined strength level is required for proper formability. Therefore a final heat treatment, in particular for hardenable steels or age-hardenable aluminium alloys is performed. One of the challenges for micro components is the way of handling and loading the small and sensitive components. The conventional way of loading for example on racks is unsuitable for small micro components,[3] because of the size and the high mounting effort. Feed baskets – generally used for components with a size of some millimetres[4,5] – are also inappropriate, because an extremely closemesh would be necessary to bear the micro components. Besides, due to the intense mechanical interaction between the sensitive parts at high temperatures the deformation probability would be very high. Furthermore, as a result of contact diffusion the threat of welding effects for the components would be high, too. To avoid these effects a new heat treatment process was developed by which the micro components are austenitized when dropping through a vertical tube furnace and are finally hardened by quenching in water respectively ambient air.[6] The characterisation of the hardened microstructure is done by metallographic analysis and ultra-micro hardness measurements.

2 MATERIAL AND METHODS

The material for the hardening experiments had to fulfil several requirements. First of all it had to be commercially available in a thickness below 100 µm to match the internal definition of micro sheets set by the CRC 747. Besides this a carbon content close to 1 mass-% was favoured to be able to use a comprehensive set of material data of the bearing steel grade 100Cr6[7,8] for thermal and transformation simulations. The steel grade C100 (steel grade EN 10132-4 resp. DIN 1.1274, AISI 1095), as a 50 µm thick foil, meets the requirements and is therefore suitable for the hardening experiments in the drop-down tube furnace. The chemical composition of the material is listed in Table 1.

Table 1. Chemical composition of the examined steel grade C100 (EN 1.1274, AISI 1095), data gained by ladle analysis of supplier

	Fe	C	Mn	Si	Cr	P	S	Ni, Mo, Ti, Al, N, V
mass.-%	Balance	1.01	0.43	0.26	0.18	0.011	0.001	not specified
EN 10132-4	-	0.95 – 1.05	0.30 – 0.60	0.15 – 0.35	< 0.4	< 0.025	< 0.025	Ni < 0.4 Mo < 0.1

The microstructure and hardness of the material in the as-received condition was unsuitable for the cold forming process because it was quenched and tempered by the supplier to a hardness level of 4908 ± 196 HM 0.05/5.0/1.0 [N/mm^2] (converted into Vickers hardness 645 ± 31 HV 0.005). In order to receive a good cold formability the C100 coil was soft-annealed with a resulting hardness of 2196 ± 241 HM 0.05/5.0/1.0 [N/mm^2] (converted 254 ± 45 HV 0.005) and a microstructure consisting of ferrite and globular as well as lamellar cementite (Figure 1). The annealing treatment was performed in a vacuum furnace at 800°C (2 h), followed by a cooling step down to 690°C with 0.1 K/min, then holding the temperature for 1h at this level and finally a very slow cooling in the furnace to room temperature.

Figure 1. Initial microstructure of C100, soft annealed, Nital etch (3%) for 25 s.

Micro components in a bowl like shape were then cold formed with a resulting diameter of 1 mm and a height of approximately 0.5 mm. Figure 2 shows an unetched cross-section of the geometry.

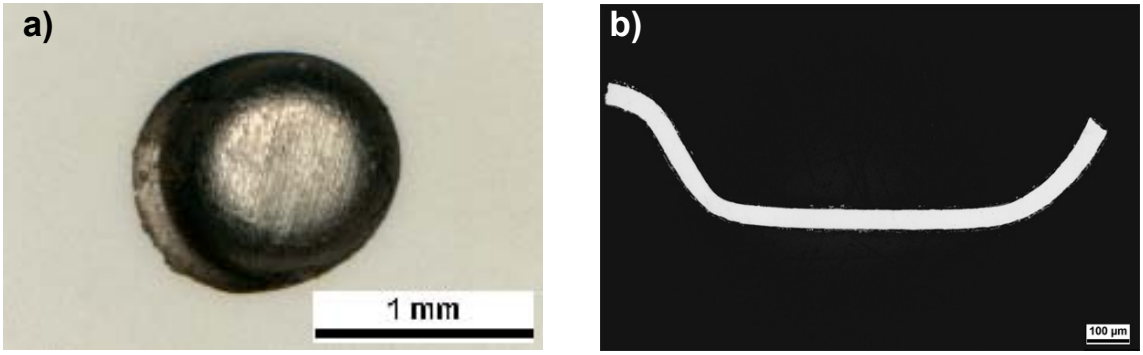

Figure 2. Bowl made of C100 (Ø 1 mm), a) photography and b) unetched cross-section.

After cold forming the micro components were heat treated in the drop-down tube furnace which has a height of 6.5 m, with a tube length of 5.5 m. The heated length is about 5 m and divided into six individually controllable heating zones with a maximum furnace temperature of 1300°C.[6] During the experiments the tube is purged with an inert gas, in this case Argon, because at temperatures above 1000°C Nitrogen as protective gas gets reactive in combination with a steel surface.[9]

Micro components were austenitized at furnace temperatures of 1000°C, 1100°C, 1200°C and 1300°C in the drop-down tube furnace in approximately 3.5 seconds falling time[6] and hardened by quenching in water. The resulting microstructure was characterised by metallographic analysis and ultra-micro hardness measurements regarding the medium hardness and the distribution of hardness values across the material thickness.

3 RESULTS

The microstructure of the micro bowl has not changed significantly during cold forming as Figure 3 illustrates in comparison with Figure 1. Polished micrographs were then used to perform the ultra-micro hardness measurements in two different ways. The mean hardness of the bowl was 2092 ± 110 HM 0.05/5.0/1.0 [N/mm^2] (converted 270 ± 26 HV 0.005) and was measured using a relatively high load of 50 mN. In a second step line scans were performed at three different positions to evaluate the hardness distribution of the bowl (Figure 4) in areas with different deformation degrees.

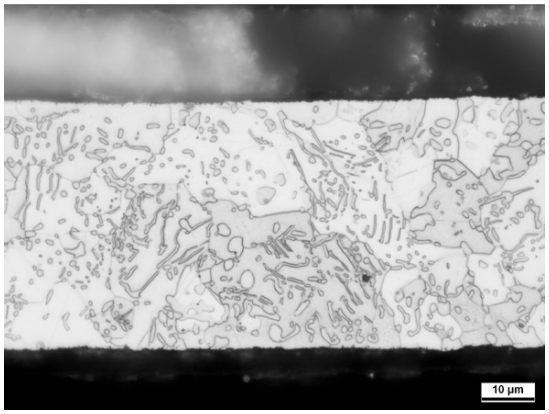

Figure 3. Microstructure of the bowl after cold forming, Nital etch (3%) for 25 s.

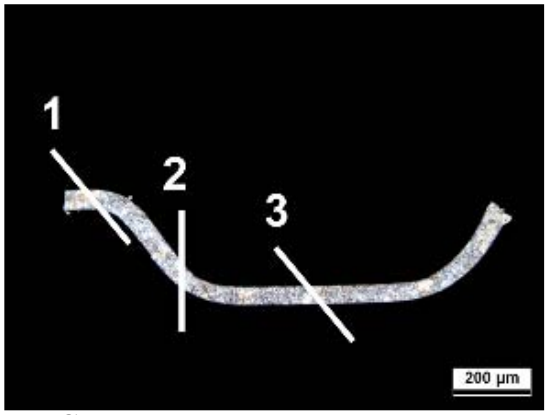

Figure 4. Bowl made of C100 (Ø 1 mm), etched cross-section, with positions of hardness distribution measurements.

Figure 5 illustrates the results of the line scans. The hardness values scatter around a hardness of 2500 HM 0.01/5.0/1.0 [N/mm^2] (converted ~300 HV 0.001) at all three positions with a tendency to higher values at position 1 and 3. On the other hand the deviation between the single values at position 2 is relatively small compared to the other two positions. Besides this a decrease of the hardness in the inner and outer surface layer is observable for position 2 and 3.

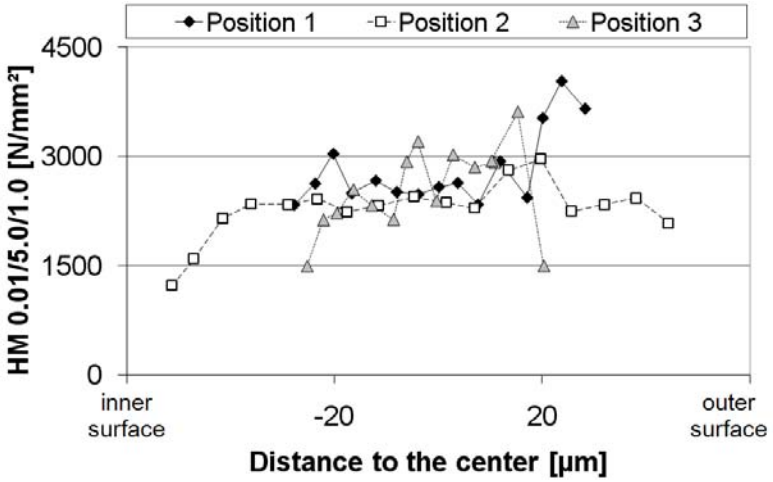

Figure 5. Hardness distributions for the cold formed bowl.

The micrographs for the hardened bowls are presented in Figure 6 and show a very homogeneous martensitic microstructure independent of the austenitizing temperature. At higher austenitizing temperatures the microstructure gets coarser as well as more defined regarding the present phases compared to the lower temperatures. Furthermore thin oxide layers are present.

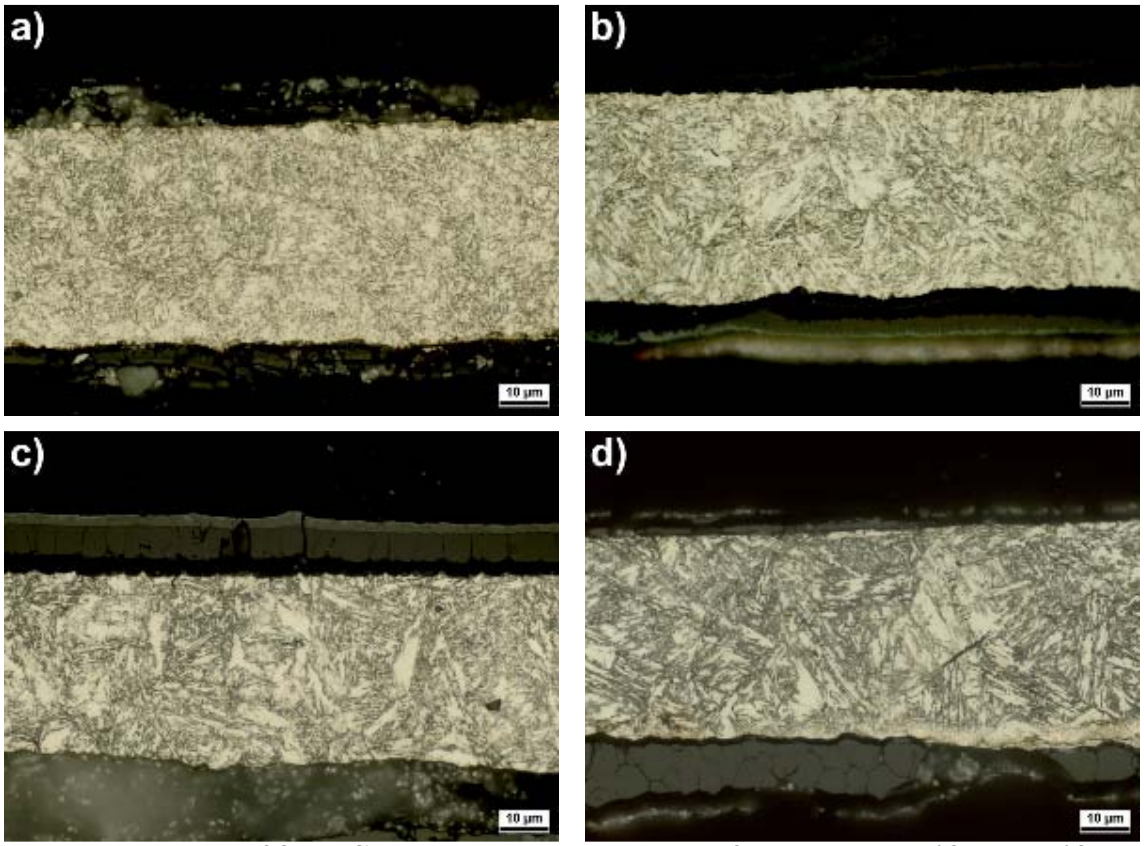

Figure 6. Bowl made of C100 (Ø 1 mm), austenized in drop-tube furnace at a) 1000°C, b) 1100°C, c) 1200°C and d) 1300°C, water quenched, Nital etch (3%) for 25 s.

The results of the hardness measurements regarding the mean hardness are presented in Figure 7.

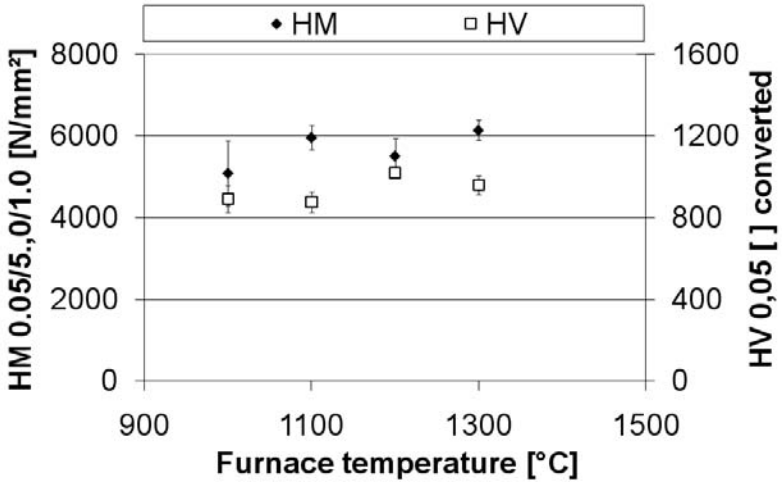

Figure 7. Results of the ultra micro hardness measurements of the bowls, austenitized during falling and water quenched.

A hardness of 5081 ± 800 HM 0.05/5.0/1.0 [N/mm^2] (converted 892 ± 67 HV 0.005) could be reached for the lowest austenitizing temperature of 1000°C. With an increasing drop-down tube furnace temperature, the mean hardness

increases as well up to 6140 ± 245 HM 0.05/5.0/1.0 [N/mm^2] (converted 959 ± 48 HV 0.005) for 1300°C. Besides this the standard deviation is reduced for higher furnace temperatures.

Figure 8 illustrates the results of the line scans. The maximum hardness is around 7000 HM 0.01/5.0/1.0 [N/mm^2] (revalued ~1100 HV 0.001) for all four evaluated drop-down tube furnace temperatures. Besides this a decrease of the hardness in the direction of the inner surface is detectable for all temperatures.

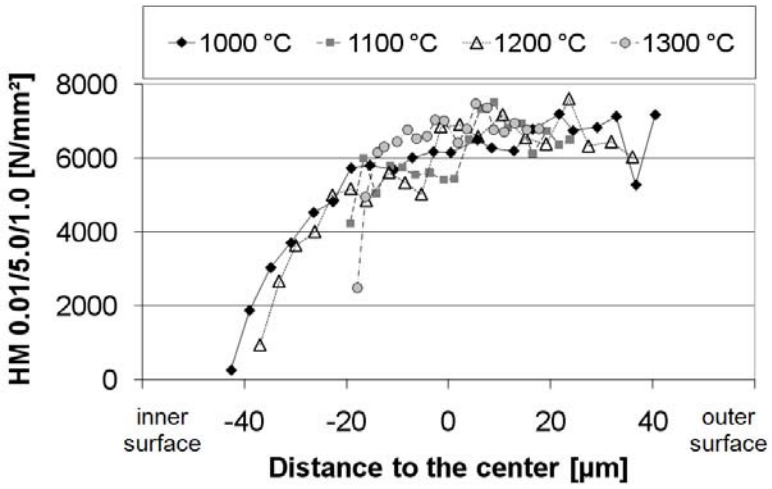

Figure 8. Hardness distributions for the bowls, austenitized during falling and water quenched, depending on the furnace temperature**.**

4 DISCUSSION

The Martens hardness value of the soft-annealed sheetbase material is a little higher than the one for the cold formed bowl, which is curious regarding the expected strengthening effect of strain-hardening. In contrast to this the Vickers hardness for the same samples is higher for the bowls, which is in accordance with the strain hardening effect. A reason for the lower Martens hardness value could be the high standard deviation (± 241 HM 0.05/5.0) of the base materials hardness value. In general the standard deviations of ultra-micro hardness measurements are higher than the ones for micro or macro hardness measurements because of the lower loads and therefore a very localized testing of perhaps only one grain or carbide. An explanation for the contrary behavior of the Martens and the Vickers hardness is the measuring situation. The Martens hardness detects the deformation under load and includes elastic as well as plastic portions. Whereas the Vickers hardness is calculated via the area of the indentation after the test load is released. Therefore it is only based on the plastic deformation and the ratio of a Martens value divided by its converted Vickers value is heavily dependent on the elastic recovery behaviour of the material.

As formerly mentioned the strain-hardening effect of the micro-cold formed bowl is not clearly detectable for the mean values either for the hardness distribution at different positions. It was expected to have a higher hardness at position 2 because of the highest deformation degree at this point. This is not the case and the hardness is even lower compared to the other two positions. All together these results lead to the assumption that micro-cold forming processes behave differently than macro cold forming ones and include other or modified effects.

Regarding the microstructure of the hardened bowls, it is very homogeneous which indicates a homogeneous hardness distribution as well. Contrarily the results of the line scans illustrate a decrease towards the inner surface of the bowl. One explanation could be that the local cooling rate is lower which would lead to diverse phase transformations and a different microstructure. Regarding the very small wall thickness this effect however is not very probable. Another possibility could be surface reactions during austenising e.g. as decarburisation. Nevertheless both should be visible in the micrographs. Therefore other reasons must be valid that are still unknown up to now. Further investigations answering this question are mandatory and ongoing. The present oxide layers indicate that the inert atmosphere was not completely established and needs to be improved.

5 CONCLUSION

The quench hardening of micro components, less than a cubic millimetre in size, could be realized in a drop-down tube furnace. Important requirements like a contact free heat treatment to avoid deformations or an oxidation free environment were realized in the developed heat treatment facility. The hardness of the cold formed bowls made of the steel grade C100 could be increased from 2196 ± 241 HM 0.05/5.0/1.0 [N/mm^2] (converted 254 ± 45 HV 0.005) up to 6140 ± 245 HM 0.05/5.0/1.0 [N/mm^2] (converted 959 ± 48 HV 0.005) in less than 4 seconds of heat treatment time. Hardness distributions across the thickness of the bowls illustrate a controversial behaviour that has to be explained in future works. Nevertheless the presented technique offers a beneficial opportunity for the heat treatment of very small components made of hardenable steel.

Future tasks cover the optimisation of the initial microstructure to improve carbide dissolution, the optimisation of the quenching techniques as well as the realisation of short term tempering of the hardened components with the tube furnace. The present quenching technique is characterised by a high component velocity at the inlet of the quenching device. The undamped strike on the water surface respectively the container bottom leads to an unintentional damage of the micro components. In order to take steps against this problem a quenching technique published by Stratton[10] shall be picked up, whereby the components can be quenched and decelerated in an opposing gas stream simultaneously.

Acknowledgment

The authors gratefully acknowledge the financial support by DFG (German Research Foundation) for Subproject A2 "Heat Treatment" within the SFB 747 (Collaborative Research Center) "Micro Cold Forming - Processes, Characterisation, Optimisation".

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