FINAL REPORT

MODELLING AND COMPLEX EXPERIMENTAL EVALUATION OF TEXTURE DEPENDENT SOLID PHASE REACTION IN METALLIC SYSTEMS, K 119655

The solid-state processes were investigated in metallic systems in course of which they take place the developed crystallographic orientation plays a crucial role in the properties and the application of the product, e.g., non-conventional texture in aluminium alloys, static and dynamic recrystallization, martensitic transformations in ferrous alloys. The novelty of research that the crystallographic anisotropy (texture) is interpreted from nano to mm size range using a combination of well-known methods during the study of the phenomenon.

Another goal was to create a virtual expert base by bringing together Hungarian orientation research experts. The research was carried out with a large number of researchers with the participation of 3 senior and 3 young researchers, as well as several PhD and graduate students. During the implementation of the project, new participants were added to the research due to the departure of some staffs. Péter Pekker left for Veszprém, so the TEM examinations and the orientation mappings were not completed, despite the large number of measurements. The research tasks of some young colleagues changed, so he left the project (Ádám Filep, Tamás Mikó), replaced by new colleagues (Máté Szűcs, Arjun Talgotra). The graduate student completed his studies and therefore became a researcher (Máté Sepsi) in his place a new student was applied (Péter Szobota). Personnel changes were approved by the supporter in all cases. The costs also had to be adjusted due to the pandemic caused by Covid 19, the conferences were held online, the costs allocated for participation were reallocated. The implementation of the research tasks was endangered by the destruction of the radiation source in the XRD device suitable for texture analysis, which repair was covered by aggregating residual costs. Cost transfers were allowed. During the research period, 2 PhD dissertations (Máté Szűcs, Adrienn Hlavács) and 3 habilitations (Jurij Sidor, Péter Barkóczy, Márton Benke) were prepared in connection with the topic. Jurij Sidor also earned his academic doctorate, and Peter Barkóczy has appointed as a professor too.

Project activities focus on 3 main areas: development of new methods and procedures in research and industrial application, physical experiments, and modelling. The results are presented accordingly. The tasks formulated in the research plan and described in the work plan were fully performed.

DEVELOPMENT OF NEW METHODS AND PROCEDURES

An XRD-NDTT (XRD based non-destructive/sampling-free) macrotexture examination method was developed. The method is based on calculating the tilting angles of centreless X-ray diffractometers which are required to realize those directions (diffraction vectors) that are used during conventional pole figure measurements. It was shown on multiple samples and an industrial-scale (full width of the rolled coil) aluminium sheet manufactured by ARCONIC Ltd. that the developed (sample cutting-free) method gave the same results with conventional texture measurement [1][2]. The theoretical background of the method was presented in conferences and manuscripts [3][4][5][6]. Instances of applications of the method were also discussed [7].

A research report won 1st ranking at the Institutional Students' Scientific Research Conference /TDK/ [8] and a patent was submitted [1]. The truly non-destructive nature of the method was exploited during the examinations of the Perfume Box of the Seuso Treasure. It was revealed that the side of the Perfume box has a notable crystallographic texture, while the bottom is texture-free [9]. Another archeologic application of the method was on the examination of silver buckle [10]. The method was also applied on silver model objects [11]. An MSc degree was obtained from the topic (Máté Sepsi) [12]. The application of the XRD-NDTT method was also applied on cross-rolled and heat treated ultra-high purity niobium specimens, which is the base material of a superconductor component of the High Luminosity Large Hadron Collider (HL-LHC) of CERN. Results show that the texture developed during cross-rolling remained after the heat treatment process, thus high-angle grain boundary movement involved recrystallization did not occur [13][14][15].

Besides developing the non-destructive texture measurement method, a simple method was developed to predict the type and relative magnitude of earing. The principle of the finalized method is to transfer the data points of {h00} pole figures of Al into the sample coordinate system and establish the vertical projections of the intensities on the rolling plane. The method was validated on a sample series taken along the width of an industrial 3xxx Al sheet both in cold rolled and annealed states. The obtained results showed a good correlation between predicted and measured average earing [16][17]18][19][20][21][22]. It was proven that the method can be applied on a wide range of sheet thicknesses that typically appear as final thickness in the industrial area. Also, it is suitable in case of special rolling procedures, such as cross-rolling [23][24]. It was shown that with the developed method, the transition of texture from rolled to recrystallized can be monitored as a function of process parameters (annealing time), thus, methodical instructions based on the thermomechanical results, that can be utilized by the industrial members were determined [21]. This method was developed to the {hhh} pole figures as well [25]. A habilitation was obtained from the topic (Márton Benke). Later on, the method was validated on hot-rolled aluminium sheets as well. It was shown that with the developed method, earing can be predicted on hot-rolled sheets, of which deep drawing cannot be performed due to their large thickness [26][27]. A PhD degree was obtained from the topic (Adrienn Hlavács). The earing prediction method was developed to BCC metals as well and validated on a samples of ferritic steel sheets [28].

In the field of high strength steels showing TWIP/TRIP behaviour, a texture evaluation method was developed which can be used to determine the directions, in which the mechanically induced $\gamma \rightarrow \alpha'$ and $\gamma \rightarrow \varepsilon$ martensitic transformations occur in the case of uniaxial tensile loading. This is based on differentiating between intensities measured in the different (namely, $\chi = 0°$ and $\chi \approx 70°$) directions of the of $\gamma\{111\}$, $\alpha'\{110\}$ and $\varepsilon\{002\}$ pole figures. Thus, unlike the EBSD technique, or the ODF representation mode of texture, the developed method differentiates between the members of the $\gamma\{111\}$ habit planes within the same unit cell. As a result, it was shown that at higher temperatures, the $\gamma \rightarrow \alpha'$ transformation occurs on the $\gamma\{111\}$ planes of which the plane normals are oriented ~70° with respect to tensile direction, while at lower temperatures, on all available $\gamma\{111\}$ planes. It was also shown that mechanical induced ε martensite only forms on these planes. The results are in good agreement with the phenomena of variant selection and the calculated mechanical driving force of the martensitic transformations [29][30]. For comparison, sample preparation method was developed for the EBSD examination of the examined steels and EBSD and TEM texture investigations were conducted. The observed results are in a good agreement with the performed EBSD and TEM orientation mapping results: the Kurdjumov-Sachs (K-S) and Shoji-Nishiyama (S-N) correlations between γ, α' and ε phases were revealed. However, it was revealed that beyond the K-S and S-N relations, the effect of martensite variant selection associated with the γ→ε and γ→α' transformations can only be revealed by the proposed method. Based on these findings, the direction of the γ→α' and γ→ε martensitic transformations and consequently, the TWIP/TRIP effect can be determined which can lead to improvements in designing energy absorbing components within the vehicle industry.

PHYSICAL EXPERIMENTS

The thermomechanical treatments of TWIP/TRIP steels resulting γ<111> fibre texture required for the investigations were performed. Namely, uniaxial tensile tests were performed at different temperatures (from room temperature to 200°C) in a climate chamber on TWIP/TRIP steels with different Cr content. The correlation between crystallographic texture and martensitic transformations was examined by conventional XRD, non-destructive XRD, EBSD and TEM orientation mapping (TEM-OM). Thus, crystallographic texture examination was realized on both macro and microscale. TEM-OM and metallographic examinations confirmed the Kurdjumov-Sachs (K-S) and Shoji-Nishiyama (S-N) correlations between γ, α' and ε phases. The complex investigation of crystallographic macro/micro texture and microstructure of the present phases was performed. It was revealed that for both austenite and ε martensite phases, the fiber/transformation texture strengthens with increasing tensile test temperature. It was concluded to be due to the different ratio of strain/transformation induced ε martensite. The characteristics of the so-called (PLC) Portevin Le-Chatelier effect was investigated during the thermomechanical treatments of TWIP/TRIP steels with different Cr content. Uniaxial tensile tests with different strain rates were performed at different temperatures. The serrations on the stress-strain curves were separated from the true stress-true strain curves after which their characteristics (amplitude, frequency) were analysed. It was shown that the strain, at which serrations initiate - called critical strain - increase with temperature and decrease with deformation rate. That is, negative strain rate sensitivity was revealed in the examined TWIP/TRIP steels [31]. The asymmetry of the avalanche-like motion of the martensite/austenite interface during thermally induced martensite↔austenite transformations was also examined. It is shown that for TWIP steels (unlike thermoelastic alloys), AE activity (thus, the number of interface steps) was considerably larger for heating than cooling [32].

Another important activity is related to a research work and developments done by CERN. This topic involved physical modelling within we cold formed the Nb sheets to such a state that characterize a complex shape structure (RFD cavity) made by Nb deep drawing at the level of mechanical test. By using cross-rolling we modified the state of the sheet material from the initial soft to a deformed state, while a defined thickness reduction was obtained by varying the rolling direction after each pass [33]. Our colleagues monitored the changes in the microstructure, while the mechanical- and non-destructive test of rolled materials was performed at CERN [15].

The texture change of 3xxx type Al sheets during semi-industrial cold rolling and subsequent annealing heat treatments was investigated as a function of composition and process parameters (temperature, holding time, type of rolling: conventional-unidirectional, special-cross rolling) of thermomechanical treatments. It was shown that increasing Mn content decreases texture in cold rolled state and promotes recrystallization process during annealing. At lower temperatures

(~190°C), only recovery occurs, while recrystallization initiates at ~280°C. Also, with the use of intermediate annealing, the final texture can be decreased. The predicted earing obtained by the method described earlier was also calculated and its variation was in a good agreement with the texture change [34][35][36][37]. A research report was prepared about the applications of the newly developed method which was ranked first at the Institutional Students' Scientific Research Conference /TDK/ [34]. An MSc thesis was also prepared from the topic (Dániel Pethő) [36]. The physical reproduction of rolling, which was used for finite element (FE) analysis of 6016 type aluminium alloy was carried out [2].

MODELLING

Thermo-mechanical tests were performed with a Gleeble physical simulator with the range of 0-1.5 logarithmic strain, 0.5 - 50 1/s strain rate and 350-500°C temperature, while time, temperature, velocity of the forming was controlled. Here, the effect of friction can be reduced but not eliminated. After the processing of upsetting test data obtained, the parameters of constitutive equation for monotonic deformation were determined [38]. After knowing the flow stress equation, it is possible to describe continuum-mechanical and material structural processes taking into account the plastic deformation history as well.

In multi-pass rolling process, the material structure changes significantly also in the non-forming process stages whose duration may be larger even with a several orders of magnitude than the time needed for effective forming. To investigate these conditions, multistage compression tests were performed with the same Gleeble simulator to see the effects of the intermediate time on the material softening. By varying the strain in the first upsetting, we were able to determine a general relation of softening occurs between forming steps, in which the effective strain played an important role that includes the strain after softening, and thus the accumulated strain energy. After knowing the initial value helps us to consider the softening of material indirectly by correcting the originally used specific flow stress curves for next passes of rolling simulation. a simplified calculation method was developed which allows to determine the characteristic parameters of velocity and deformation field of cold-rolled material and evaluate experimental rolling processes. It also analyzes the 3D material flow when a significant widening of material occurs. Experimental rolling was analyzed by finite element calculation similarly. After comparing the two models, we found that the mechanical fields differ slightly. The simplified calculation procedure is also suitable for testing multi-pass rolling and it also provides input data of the texture calculation (velocity gradient tensor of a selected material point) [39][40]. Texture simulation was performed by using VPSC code including the so-called viscoplastic self-consistent method. Some measured data of rolled sheet obtained by X-ray diffraction will be used as input data for the texture simulations [41]. An analysis on modelling of cold rolling of 6016 type aluminium alloy was also performed by means of mean-field and full-field approaches. For the intra-grain deformation phenomena, the crystal elasto-visco-plastic FE model was used. The deformation history was calculated with analytical approaches and a FE model with isotropic mechanical properties, which accounted for various degree of accuracy. It was shown that the applied analytical approximations accoupled with the crystal plasticity model employed can carry out texture simulations close to the one performed with the crystal plasticity model with the deformation history obtained by means of the FE model [2].

Analysing the hot rolling's effect on microstructure was revealed that the recovery has a significant effect to the recrystallization process. For modelling the crystallographic changes, cellular automata (CA) were employed. The Euler angles were assigned to the grains. The twin boundary misorientation is calculated based on the Euler angles. The model simplifies the tilt/twist position of the grain boundary to take into consideration the rotation of the <111> directions. This model gives the appropriate grain boundary character function [42]. A research report was prepared, and the second place was won at the National Students' Scientific Research Conference /OTDK/ [43]. The calculation of the recrystallized volume fraction is based on Johnson-Mehl-Avrami-Kolmogorov's (JMAK) time – transformation ratio, and DSC measurements. The evaluation of the mechanical tests revealed that the same kinetic constants can be used in the dynamic description as in the static phenomenon [44][45][46][47][48][49][50]. For studying the recovery, another automaton was employed. The effect of the strain hardening to the stored energy was evaluated by DSC tests. The tests were made on sheet samples which were rolled by different reductions. Recrystallization was not occurred in the samples. The deformation path of the developed simulation was set to similar as the compress tests. The developed simulation then extends to a whole rolling process of a 450mm thick slab. Using the cellular automaton, the effects of mainly the changes of the sequence of reductions and rolling temperatures were studied [51][52][53].

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