

Rapid alloy prototyping: Compositional and thermo-mechanical high throughput bulk combinatorial design of structural materials based on the example of 30Mn–1.2C–*x*Al triplex steels

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Abstract

We introduce a new experimental approach to the compositional and thermo-mechanical design and rapid maturation of bulk structural materials. This method, termed rapid alloy prototyping (RAP), is based on semi-continuous high throughput bulk casting, rolling, heat treatment and sample preparation techniques. 45 Material conditions, i.e. 5 alloys with systematically varied compositions, each modified by 9 different ageing treatments, were produced and investigated within 35 h. This accelerated screening of the tensile, hardness and microstructural properties as a function of chemical and thermo-mechanical parameters allows the highly efficient and knowledge-based design of bulk structural alloys. The efficiency of the approach was demonstrated on a group of Fe–30Mn–1.2C–*x*Al steels which exhibit a wide spectrum of structural and mechanical characteristics, depending on the respective Al concentration. High amounts of Al addition (>8 wt.%) resulted in pronounced strengthening, while low concentrations (<2 wt.%) led to embrittlement of the material during ageing.

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1. Introduction

Increased efficiency in the development of novel metallic structural materials is a key to providing fundamental and applied alloy solutions in the fields of energy, mobility, health, infrastructure and safety. Examples of the associated basic metallurgical questions are alloy- and processing-sensitive changes in complex strain hardening phenomena. In high strength steels these are often associated with displacive transformations, such as twinning and transformation-induced plasticity (TWIP and TRIP), which can be tuned through variations in the stacking fault energy and microstructure. Associated engineering issues are systematic analysis of the corresponding trends in

texture evolution, sheet forming and damage parameters, joining behaviour, hydrogen susceptibility or fatigue.

In thin film systems, in which combinatorial high throughput methods were invented and established, substantial progress has been made in a number of materials design fields, such as shape memory alloys, MAX phases and shape change materials [1–6]. Such combinatorial methods have mostly been applied to the design of functional materials, where, typically, the intrinsic composition-dependent properties dominate over the relevance of the microstructure. In structural alloys, on the other hand, the relevant length scales that determine the mechanical behaviour are parameters such as grain size, texture, precipitate dispersion and topology and the dislocation cell structure, to name but a few. The correlation lengths associated with such lattice defects are usually of the order of several nanometres to multiple micrometres, hence, they usually exceed the dimensions accessible in thin films. This

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means that the mechanical properties of structural materials are not only determined by their chemical composition, but to a large extent by the microstructure, which in turn is greatly influenced by the thermo-mechanical treatments (TMTs) applied after synthesis [7–9].

Consequently, the current conventional approach for the experimentally guided development of metallic structural materials typically consists of a number of iterative loops that include the following basic steps: Bulk casting of a single composition charge, hot and/or cold deformation (e.g. conventional or thermo-mechanical rolling), heat treatment (such as quenching, partitioning or ageing), machining of tensile specimens and mechanical testing. Coupled with the wide range of possible chemical compositions and different TMT setups, this kind of incremental trial and error approach typically provides robust mechanical data on novel alloys [10,11], however, it is often too time consuming for a more efficient investigation and the maturation of complex alloy systems as a function of composition, TMT processing and microstructure.

Thus it becomes clear that the development of novel and more efficient high throughput methods for the bulk synthesis, TMT processing and following investigations of structural materials is of great interest. It is aimed at drastically increasing both the scientific and engineering efficiency by reducing the time between an alloy design idea and final evaluation of the material's mechanical and microstructural properties from several weeks or even months down to hours. Materials exhibiting desirable properties (“hits”) can be readily selected from the investigated composition/TMT matrix and then scaled up via conventional methods for more detailed investigations based on the information collected. Rapid and systematic screening of prototype alloys and TMT setups, ideally employed in concert with theory-based, alloy-sensitive simulation methods [12–15], thereby allows the efficient and knowledge-based design of structural materials.

Steels containing high amounts of manganese (Mn), aluminium (Al) and carbon (C) were here chosen as an example material system to utilize the novel approach outlined above. Also referred to as “Triplex” steels, these materials exhibit a low mass density, high strength and excellent ductility in comparison with established steels for structural engineering applications [10,16,17]. The authors derived this attractive property profile from an austenitic or austenitic/ferritic matrix, both stabilized and strengthened by alloying iron (Fe) with Mn (18–28 wt.%) and C (0.7–1.2 wt.%), together with Al addition (3–12 wt.%) for low specific weight and improved corrosion resistance [10,18–20]. Profound changes in the mechanical properties can be achieved by ageing the material after solution annealing and quenching. More specifically, κ -Al(Fe,Mn)₃C carbides were found to precipitate from the matrix during ageing, growing from C-enriched areas, most probably formed via spinodal decomposition during quenching [17,21,22]. The further development of Fe–Mn–Al–C alloys must involve simultaneous investigation of the respective micro-

structural phenomena by high resolution characterisation techniques such as atom probe tomography (APT) and transmission electron microscopy (TEM). Such techniques, however, require increased efforts in sample preparation and experimental procedures [23–26] and are, therefore, preferably applied only to those compositions and microstructures that reveal the most interesting characteristics or properties. The wide range of possible chemical compositions in the Fe–Mn–Al–C quaternary system, combined with a large matrix of possible TMT routes (especially ageing time and temperature), justifies the high level of motivation for the development and use of rapid alloy prototyping (RAP) techniques as an efficient tool for structural materials development.

2. Objective

The objective of this work was to present a new approach to the rapid investigation of bulk metallic structural materials, whereby the mechanical properties of a group of alloys systematically varied in terms of chemical composition and the imposed TMT treatments can be evaluated simultaneously and thus with a higher throughput than with conventional methods and step by step iteration of these parameters. The approach is referred to as RAP. As an example here we study a novel group of 30Mn–1.2C steels in terms of the effects of varying Al content and different ageing conditions on the microstructure and properties. Selected results are directly compared with data obtained from conventionally synthesised and processed samples to validate the approach.

3. Materials and methods

3.1. Production of samples

Fig. 1a schematically illustrates the experimental process of our RAP approach, which is detailed below. The primary synthesis of 30Mn–1.2C–*x*Al (wt.%) samples was conducted via melting and casting in a vacuum induction melting (VIM) furnace (heating power 60 kW, 4 kHz, argon (Ar) atmosphere of 400 mbar). The chemical compositions of the alloys in this study (target/actual values by wet chemical analysis) are listed in Table 1. Fig. 1b illustrates the device installed inside the furnace to create the five different alloys in one casting operation. Five copper (Cu) moulds with respective internal volumes of 10 × 50 × 150 mm³ was moved step by step on a linear stage by an electric drive and successively filled with melt from an ingot of 4 kg basic capacity. After each cast, the composition of the melt remaining in the ingot was precisely altered by adding precalculated amounts of the alloying elements (in this case Al) through the furnace air-lock. Each mould was formed into a 20 mm high 45° funnel at the top for ease of casting and placed on a water-cooled Cu plate. Thus the shrinkage during solidification occurred at the top of the enlarged funnel and not as cavities within

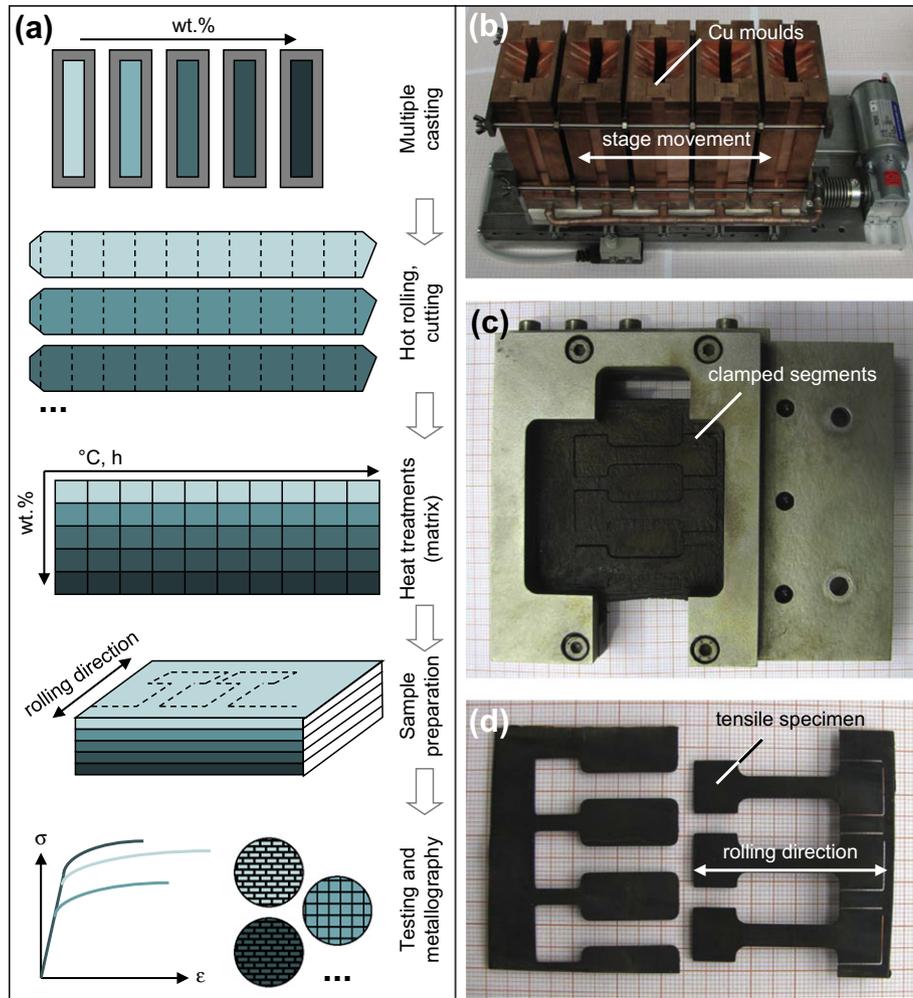


Fig. 1. Illustration of the sample production process: (a) schematic sketch of the RAP approach with the differently shaded colours referring to the different chemical compositions; (b) device for casting five alloys into separate Cu moulds placed on a linear stage; (c) five segments after completion of the heat treatment clamped together for simultaneous spark erosion; (d) one segment after completion of the spark erosion preparation of three tensile specimens. RAP, rapid alloy prototyping.

Table 1

Target (left) and actual values (right) for the chemical compositions in wt.% in the RAP experiments obtained by wet chemical analysis.

Target values (wt.%)	Actual values (wt.%)
Fe–30Mn–1.2C	Fe–30.5Mn–1.23C
Fe–30Mn–1.2C–2Al	Fe–31.0Mn–1.20C–2.12Al
Fe–30Mn–1.2C–4Al	Fe–31.4Mn–1.19C–4.30Al
Fe–30Mn–1.2C–6Al	Fe–31.3Mn–1.20C–6.42Al
Fe–30Mn–1.2C–8Al	Fe–31.6Mn–1.17C–9.04Al

RAP, rapid alloy prototyping.

the bulk. Removing the top (remove the funnel) and bottom (for chemical analysis) from the cast blocks after cooling to room temperature left five $10 \times 50 \times 130 \text{ mm}^3$ rectangular blocks.

The 10 mm thick blocks were then hot rolled (air atmosphere) at $1100 \text{ }^\circ\text{C}$ to $2 \pm 0.1 \text{ mm}$ thick and $\sim 500 \text{ mm}$ long sheets. After the last rolling pass the sheets were reheated to $1100 \text{ }^\circ\text{C}$, quenched in water and cut perpendicular to

the rolling direction into 55 mm long segments. This gave nine segments with the dimensions $2 \times 60 \times 55 \text{ mm}$ for each alloy (45 in total). Punch marking of the segments with codes for both alloy composition and the applied heat treatment ensured reliable identification of the specimens throughout the following processing steps. Homogenization of the segments was performed by annealing at $1100 \text{ }^\circ\text{C}$ for 2 h under an Ar atmosphere, followed by water quenching in clamps to minimise distortion. Ageing of the segments was performed in air at 450, 500, 550, and $600 \text{ }^\circ\text{C}$ for 0, 1 and 24 h at each temperature (0 h representing the as-homogenised state), followed by oil quenching. This resulted in a matrix of 45 different alloy/TMT conditions. Scales were removed from the surfaces by low pressure, fine grit sand-blasting after completion of the heat treatments.

Samples for mechanical testing and microstructure investigation were prepared from the segments by spark erosion. In order to increase the efficiency of this gentle but rather slow technique (cutting speed $\sim 6 \text{ mm s}^{-1}$) five

segments were clamped on top of each other and simultaneously cut, as shown in Fig. 1c. Tensile samples were prepared with a width of 5 mm (cross-sectional area 10 mm²), gauge length of 17.89 mm (equivalent to A5 proportional samples) and a longitudinal axis parallel to the rolling direction. A thin 0.2 mm bridge was left to keep the cut specimens attached to their respective segments, so that they could be identified and separated from the marked segment just before tensile testing. Three samples were prepared for each material and heat treatment condition, as shown in Fig. 1d; leftover material was used for hardness testing and metallographic preparation. RAP sample identification throughout this work follows the respective target alloy compositions in wt.% (Table 1).

For reference and validation of the RAP results tensile samples of two alloys were produced by conventional metallurgy. Single charge melts of Fe–30Mn–1.2C alloys with additions of 2 and 8 wt.% Al, respectively, were cast into a 40 × 60 × 200 mm³ Cu mould using the same VIM furnace as for the RAP experiments. The surfaces of the cast blocks were then milled and the alloys hot rolled at 1100 °C (air atmosphere) to 10 mm thickness and quenched in water. After homogenisation at 1100 °C for 2 h (Ar atmosphere) and water quenching the plates were cut by spark erosion into strips (10 × 10 mm cross-section), from which cylindrical tensile samples were lathed (DIN 50125 form B, 6 mm diameter, longitudinal axis parallel to the rolling direction). Ageing was performed in air at 450 °C for 1 h, followed by oil quenching.

3.2. Mechanical testing and characterization

Tensile testing of the RAP samples was conducted at room temperature with an initial strain rate of 10^{−3} s^{−1} using a hydraulic Instron 8511 testing machine. Hardness (Brinell HBW, ball diameter 2.5 mm) was measured with a Wolpert DIA Testor 2RC on the outer surface of the segments after grinding them with 1000 grit. All values obtained from both tensile and hardness tests represent the averages of three individual measurements for each alloy/TMT combination. Cross-sectional areas of selected samples were prepared in the plane perpendicular to the rolling direction by grinding and polishing using standard metallographic techniques. The cross-sections were investigated by optical microscopy (OM) with a Zeiss Axiophot 1.

A Zwick/Roell Z100 machine was used for tensile testing of the conventionally fabricated samples at room temperature at a starting strain rate of 10^{−4} s^{−1}. Two tests were carried out for each alloy.

4. Results

4.1. Mechanical testing of the RAP samples

Following the high throughput procedure outlined above five alloy compositions, each exposed to nine respective heat treatments, were produced, processed and

evaluated within 35 h. The mechanical properties of these 45 different material conditions (i.e. in total 135 tensile tests and hardness measurements) are shown as an overview in Fig. 2 in terms of the yield strength (YS) (Fig. 2a), ultimate tensile strength (UTS) (Fig. 2b), total elongation (TE) (Fig. 2c) and hardness (Fig. 2d). The results are plotted according to the systematically varied Al content (Table 1) and colour coded according to the individual ageing conditions. Pronounced effects associated with the changes in chemical composition and ageing parameters on the mechanical behaviour of the materials can be clearly distinguished. For the reference material (no Al addition, i.e. Fe–30Mn–1.2C) the best mechanical behaviour was observed for the as-homogenised, non-aged state. This alloy was characterised by a YS of 360 MPa, strong work hardening (UTS = 830 MPa) and high ductility (TE = 77%). Ageing of the Fe–30Mn–1.2C alloy showed that the YS was virtually unchanged and the hardness slightly increased. However, ageing greatly reduces both the UTS and TE. This embrittlement becomes most apparent for long ageing times (24 h) and higher temperatures (>500 °C). For the alloy Fe–30Mn–1.2C–8Al, i.e. the material with the highest Al concentration, the opposite trend applied. Without ageing the mechanical data are similar to those of the Al-free alloy, with only a slight change in YS (increase), UTS and TE (decrease). Ageing treatments for 1 h, however, led to a simultaneous improvement in YS, UTS and hardness (increasing with temperature) and to only a slight drop in TE. Ageing of alloy Fe–30Mn–1.2C–8Al for 24 h further increased YS, UTS and hardness to values almost twice as high as in the as-homogenized state, but also drastically reduced the ductility. The mechanical data for the alloys with intermediate Al contents appear as the superimposition of the two different behaviours described above: as a general rule, the values obtained for the alloys with 2, 4 and 6 wt.% Al lie between the respective data from alloys without and with 8 wt.% Al. The alloys with Al additions of 4 and 6 wt.% especially are only very weakly affected by the applied ageing treatments in terms of their mechanical data compared with the alloys Fe–30Mn–1.2C (weakening/embrittlement) and Fe–30Mn–1.2C–8Al (strengthening).

In order to better visualise the above described trends in the mechanical behaviour for the investigated composition/TMT matrix selected results are presented in Fig. 3. Changes in YS, UTS, TE and hardness over the ageing time at 550 °C of the alloys Fe–30Mn–1.2C, Fe–30Mn–1.2C–4Al and Fe–30Mn–1.2C–8Al are displayed in Fig. 3a–c, respectively. The experimental scatter is indicated by the error bars in Fig. 3a.

4.2. Microstructure of the RAP samples

Fig. 4 shows optical micrographs after the different processing steps for the example alloy Fe–30Mn–1.2C at different magnifications. No cracks, pores or macro-segregations can be observed after casting in the overview image on the

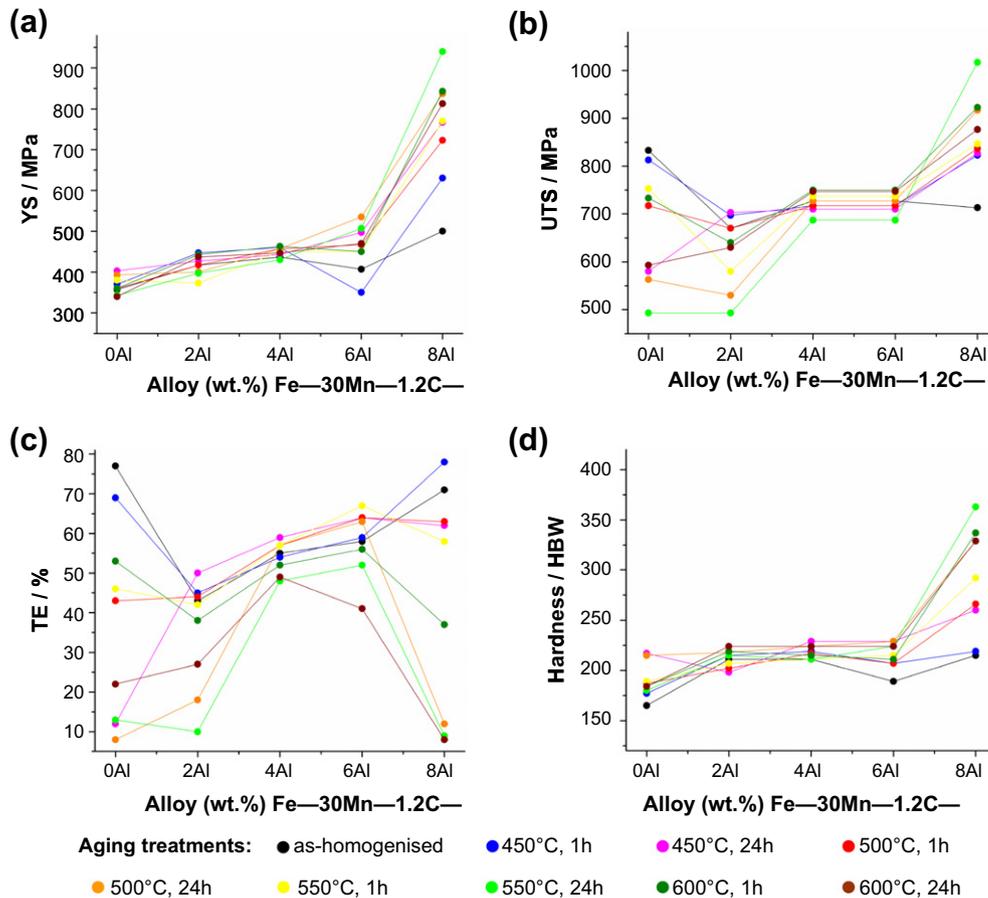


Fig. 2. Overview of the mechanical properties obtained from the RAP experiments as a function of alloy composition (Table 1) and the applied ageing treatment: (a) yield stress (YS); (b) ultimate tensile stress (UTS); (c) total elongation (TE); (d) hardness. RAP, rapid alloy prototyping.

left-hand side of Fig. 4a. The high magnification image on the right-hand side reveals a coarse dendritic microstructure that is typical of as-cast alloy microstructures. Hot rolling and water quenching (Fig. 4b) result in a fully recrystallized microstructure (grain size $\sim 20 \mu\text{m}$) with some retained micro-segregations (dark lines in the micrographs). Within this context it is important to underline that chemical homogenization can usually be better obtained via TMT involving static, post-dynamic or dynamic recrystallization, rather than by a static heat treatment alone. This is due to the fact that recrystallisation involves the motion of high angle grain boundaries, which provide much higher diffusion coefficients compared with bulk diffusion.

Optical micrographs showing the microstructure of selected samples after complete processing and heat treatment are presented in Fig. 5. Fig. 5a–c corresponds to the Fe-30Mn-1.2C-based alloys with the addition of 0, 4 and 8 wt.% Al, respectively. Microstructures in the as-homogenised state (unaged) are shown on the left-hand side of Fig. 5, those after ageing at 550 °C for 24 h on the right-hand side, respectively. In the as-homogenized state the three alloys exhibit almost identical austenitic microstructures with an average grain size of about

80 μm , few twins (increasing with higher Al content) and no apparent micro-segregations. After ageing, however, pronounced differences between the three alloys could be observed. Coarse particles with a diameter of $\sim 10 \mu\text{m}$ appear at the grain boundaries of the Al-free alloy (Fe-30Mn-1.2C), most probably consisting of a pearlitic ferrite/(Fe,Mn)₃C microstructure. The addition of Al to the alloy Fe-30Mn-1.2C-4Al apparently constrained the formation of those phases during ageing, as the number density and size of the particles was now significantly lower than in the Al-free material and only thin films appeared on the grain boundaries of the alloy with 4 wt.% Al. A further increase in Al content (alloy Fe-30Mn-1.2C-8Al) resulted in the complete absence of grain boundary particles during ageing, but a large number of unevenly distributed small particles appeared within the grains, giving them a darker contrast.

4.3. Tensile testing of the conventionally produced samples

For reference and comparison Fig. 6 shows exemplar engineering stress–strain curves for the alloys Fe-30Mn-1.2C-2Al (red) and Fe-30Mn-1.2C-8Al (blue) after ageing at 450 °C for 1 h, obtained by following the conventional

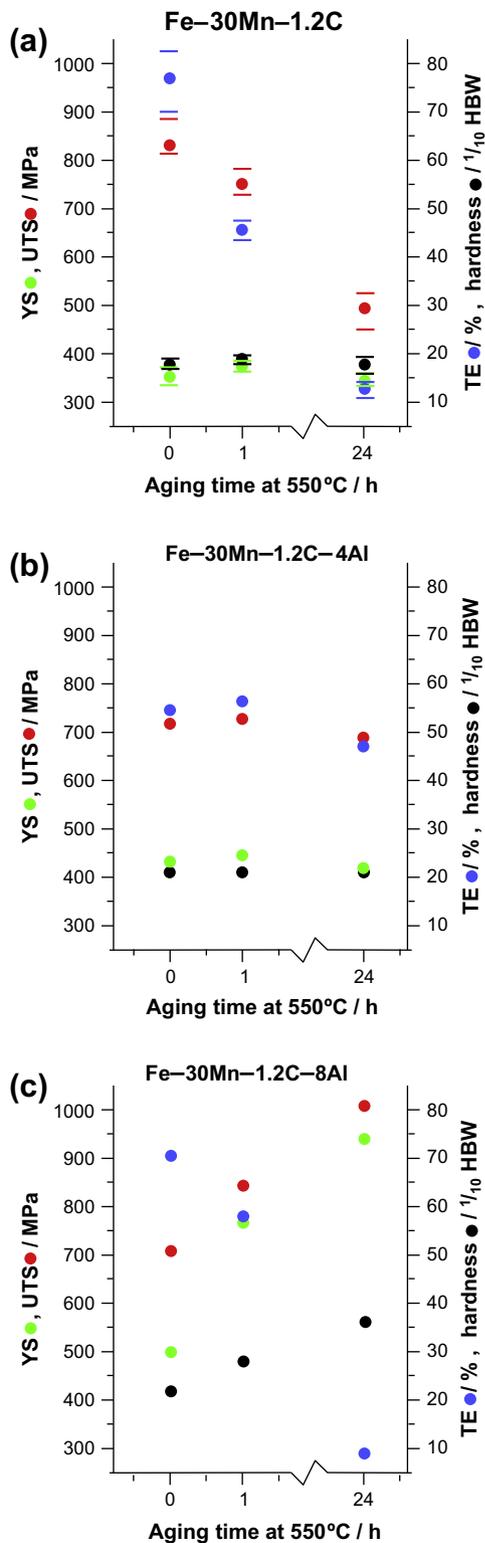


Fig. 3. Selected mechanical test results from the RAP experiments: yield strength (YS), ultimate tensile strength (UTS), total elongation (TE) and hardness as functions of ageing time at 550 °C for alloys: (a) Fe-30Mn-1.2C; (b) Fe-30Mn-1.2C-4Al; (c) Fe-30Mn-1.2C-8Al.

synthesis and processing (Fig. 6a) and RAP (Fig. 6b) approaches. For the alloy Fe-30Mn-1.2C-8Al both the RAP and conventional data are in the same range, with an only lightly increased YS (610 to 540 MPa), lower

UTS (810 to 890 MPa) and almost identical ductility (76 to 73%) of the RAP samples. While this trend (slightly higher YS and lower UTS) is the same for the alloy Fe-30Mn-1.2-2Al, the RAP samples now exhibited significantly less ductility than the conventional specimens (49 compared with 84%) under this specific material condition. The unusually large elastic strain that is apparent in Fig. 6b can be attributed to slight deformation (bending) of the RAP samples, which could not be completely avoided despite the clamping procedures applied during quenching of the thin segments.

5. Discussion

5.1. Microstructure and mechanical properties of the 30Mn-1.2C-xAl steels

The novel bulk RAP approach introduced in this work provides, for the first time, a systematic evaluation of the compositional and thermo-mechanical trends associated with a change in the Al content of a group of Triplex steels with high Mn and C concentrations. We observed that without the addition of Al to the 30Mn-1.2C steels the most favourable mechanical properties were obtained for the as-homogenised state (Figs. 2 and 3a). The observed properties are in reasonable agreement with data reported for Mn-C alloyed TWIP steels of a similar chemical composition [27,28]. The observed embrittlement during ageing can be related to the formation of the coarse, pearlitic particles on the grain boundaries [27] (Fig. 5a).

High amounts of Al (~8 wt.%), on the other hand, result in pronounced strengthening during ageing, depending on the time and temperature (Figs. 2 and 3c), and no coarse particles could be observed in this case (Fig. 5c). In the light of previous results this typical precipitation hardening behaviour, which allows tuning of the strength and ductility, can be explained by the formation and growth of κ carbides during ageing [17,18,21]. Due to their reportedly small size, which is of the order of several nanometres, the κ carbides could not be reliably detected or identified in the high throughput RAP OM observations conducted in this study. The darker particles visible in Fig. 5c might be linked to κ carbides.

Alloys with intermediate Al concentrations (about 2–6 wt.%) do not offer mechanical properties on the same level compared with the aforementioned extreme cases under their respective optimal conditions (i.e. after the respective most suitable ageing treatments). On the other hand, a much smaller influence of the ageing parameters on tensile behaviour can be observed in these cases (Figs. 2 and 3b). Within the limitations of this study (confined range of applied heat treatments, OM investigations, etc.) this improved stability of the mechanical properties during thermal exposure can be attributed to a concerted formation of κ carbides and grain boundary pearlite, balancing the strengthening and embrittlement effects of intermediate amounts of Al.

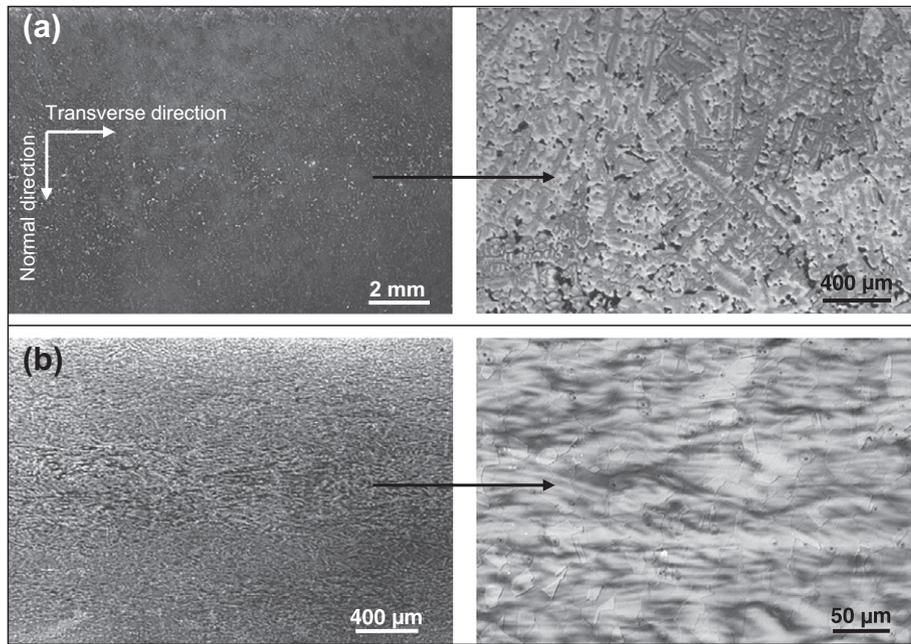


Fig. 4. Optical micrographs of the alloy Fe-30Mn-1.2C from different stages of the RAP sample production process at different magnifications: (a) after casting; (b) after hot rolling and water quenching. RAP, rapid alloy prototyping.

In general it should be underlined that detailed investigations of the role of κ carbides and pearlite particles on the deformation mechanisms, as well as the precipitation type and the structural nature of the κ carbides (i.e. spinodal vs. nucleation/growth), require higher resolution techniques, such as TEM or APT. Nonetheless, the mechanical data on both RAP (Figs. 2 and 3) and conventionally synthesised and processed alloys (Fig. 6a) are in good agreement with previously reported values for Fe-Mn-Al-C steels [17,18]. The RAP results suggest that future efforts regarding more detailed nanostructural investigations should focus on such high-C triplex steels with high Al concentrations (>8 wt.%), as they offer the possibility of covering the widest range of mechanical properties via ageing treatments (scalability, Fig. 2) and exhibit the lowest possible specific weight of all such steels.

5.2. Evaluation of the RAP approach

Following the RAP approach introduced in this work, 45 different material conditions were synthesised, processed and tested within 35 h (Fig. 1a). Investigations of the same matrix, i.e. five different alloy compositions combined with nine different heat treatments, would require a minimum of about 6 weeks when using conventional metallurgical, processing, and testing methods. For both cases the effect of the TMT parameters on the duration of such systematic combinatorial experiments, especially the ageing time, needs to be taken into account. The main key ingredients to exploiting the full potential of an accelerated investigation of bulk structural alloys via RAP are the multiple casting operation (five alloys instead of one, Fig. 1b),

simultaneous sample preparation by spark erosion (15 samples at the same time, Fig. 1c and d) and, most importantly, the efficient sequential adaptation of all processes throughout the entire chain of experiments. It should be noted that our approach is a combination of efficiently adapted but already established processing steps. This results in robust processing, i.e. less calibrating or inter-checking between steps is needed, thus ensuring fast processing and reliable data. Furthermore, the modular concept offers the possibility of replacing individual processing steps with additional or different techniques, for example exchanging synthesis via casting by powder-based processes, while maintaining the fast sequential TMT and testing procedures. The RAP synthesis via bulk casting and hot rolling applied here, however, approximates processes and microstructures which are very similar to those of structural materials produced on an industrial scale. While alternative methods of mechanical probing can be used, tensile testing appears to be the most representative method for the evaluation of the mechanical behaviour of structural materials, within the specific limitations of our approach discussed later in the text (surface effects). Alternatively, indentation experiments can, for example, reveal hardness and even certain strength measures, but they provide only a limited insight into the ductility of the investigated bulk materials. This is clearly illustrated by the ageing-induced embrittlement of alloy Fe-30Mn-1.2C in our study. The TE values decreased significantly with ageing time and temperature, whereas the hardness data showed a slight increase (Figs. 2a and d and 3a).

Some deviation in chemical composition between the target and actual values of the RAP samples (Table 1)

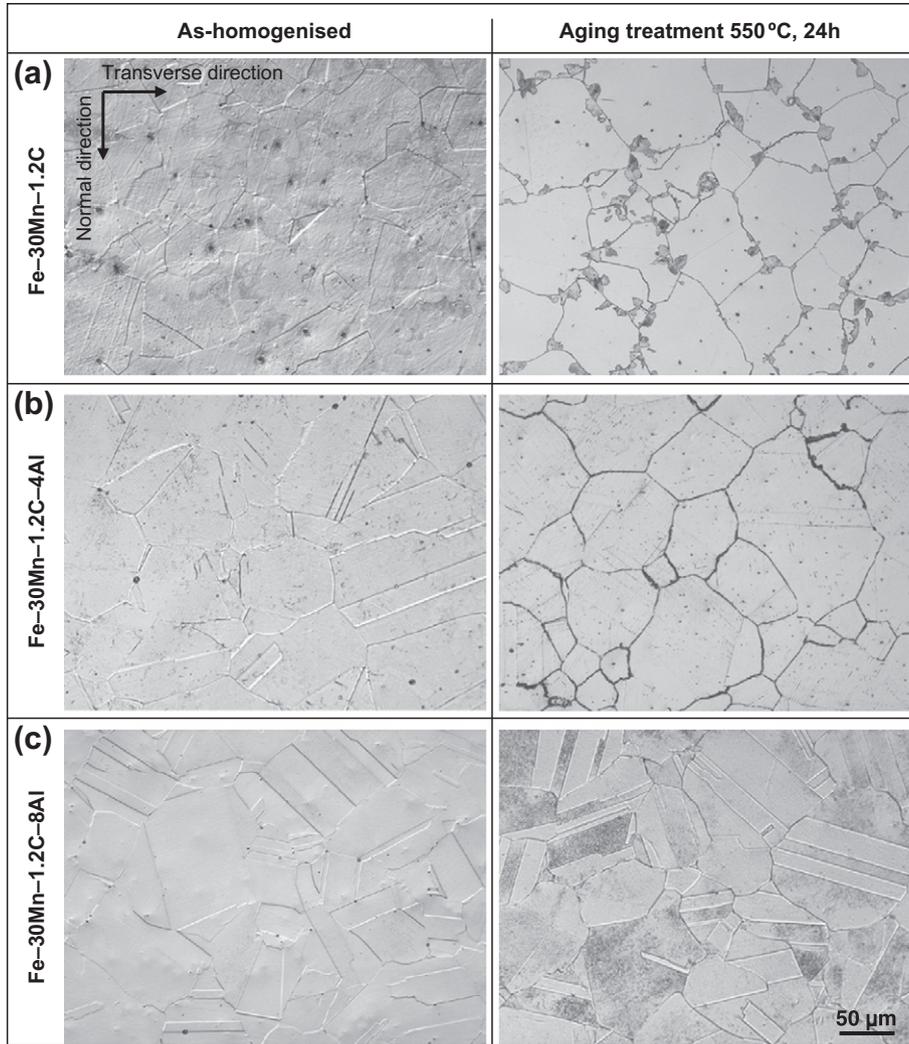


Fig. 5. Optical micrographs of selected RAP samples in the as-homogenized state (left pictures) and after ageing at 550 °C for 24 h (right pictures): (a) Fe-30Mn-1.2C; (b) Fe-30Mn-1.2C-4Al; (c) Fe-30Mn-1.2C-8Al. RAP, rapid alloy prototyping.

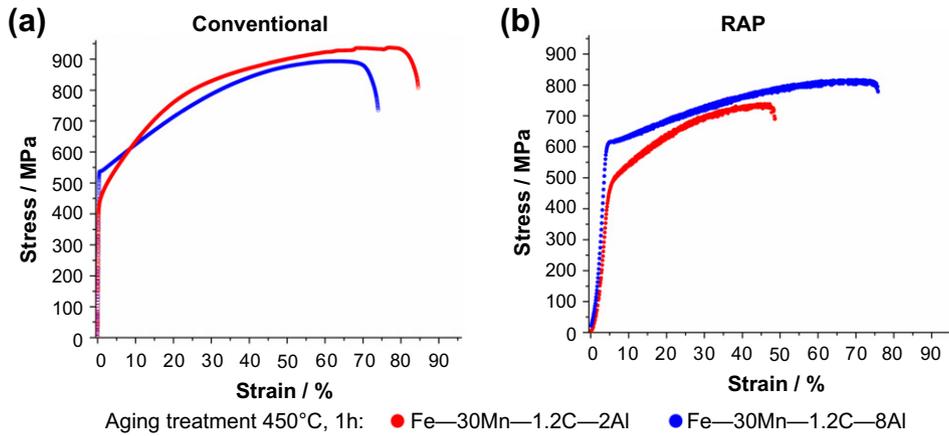


Fig. 6. Engineering stress–strain plots of the alloys Fe-30Mn-1.2C-2Al (red curves) and Fe-30Mn-1.2C-8Al (blue curves) after aging at 450 °C for 1 h: (a) conventionally synthesized and processed (single charge casting, cylindrical samples); (b) exemplary RAP samples. RAP, rapid alloy prototyping.

occurs as multiple charging is more difficult than conventional single charge casting, and the deviations naturally become larger with ongoing casting/alloying procedures.

Nonetheless, the achieved chemical concentrations are accurate enough for the intended screening of the alloy system under investigation. While the deviations in terms of

tensile properties between conventional and RAP samples are typically below 10%, the sporadically occurring strong relative drop in ductility and subsequent lower UTS in the RAP experiments (Fig. 6) are mainly attributed to surface effects. The improved surface quality (smoothness, removed scales and decarburized areas) and the more compliant geometry of the cylindrical tensile samples reduced the tendency for premature localization and rupture before the maximum strength of the materials was reached. Other influencing factors which highlight the dependency between testing procedures and the obtained values are the higher speed of tensile testing and, more importantly, the smaller thickness of the RAP samples (2 mm compared with 6 mm). As thinner specimens reach the set temperature faster than thicker ones the effects of ageing (in this case a higher YS and lower TE) will thus be more pronounced. The reasons for the larger discrepancy in ductility for the alloy Fe–30Mn–1.2C–2Al in contrast to the good agreement in the case of the alloys containing 8 wt.% Al, however, clearly requires more detailed analysis. Future investigations on a growing number of different alloy systems will result in enhanced statistics, so that the precision of RAP for screening mechanical properties can be further improved.

As our approach is designed to identify compositional, process-related and microstructural trends that are relevant to the mechanical behaviour (Figs. 2 and 3), it allows reliable pre-selection of material parameters and highlights the intended role of RAP as a complementary tool in structural materials development. The surface effects described above should leave the RAP data generally on the “safe” side, i.e. slightly improved tensile properties (especially ductility) can be expected after up-scaling a selected prototype alloy via conventional methods. It should be stressed that a basic knowledge of the investigated alloy system, via thermodynamic calculations or existing experience, remains as important as for conventional synthesis and processing of structural materials in order to select appropriate TMT parameters and alloy compositions.

6. Summary and conclusions

In this study we have introduced a new approach to achieving a higher throughput in the experimentally guided design of bulk structural materials. We used a group of 30Mn–1.2C–xAl steels (wt.%) as an example. Tensile and hardness testing as well as optical microscopy were performed on a matrix of 45 samples, i.e. five alloy compositions (0–8 wt.% Al) and nine different ageing treatments (450–600 °C for 0, 1 and 24 h). Additionally, two conventionally synthesised and processed alloys of similar chemical composition were investigated in order to validate the RAP approach. The following conclusions can be drawn.

1. Chosen here as an exemplary material system, the Fe–Mn–Al–C steels investigated exhibited widely differing characteristics, depending on their respective Al concentration. Ageing of the material without Al addition resulted in the formation of coarse pearlitic particles at the grain boundaries and subsequent embrittlement. On the other hand, pronounced strengthening could be observed after ageing the alloy with the highest Al concentration, most probably linked to the formation of small and dispersed κ -Al(Fe,Mn)₃C carbides. Intermediate Al concentrations led to less favourable mechanical properties compared with the alloy variants with no or very high amounts of Al, but exhibited a greater thermal stability.
2. Future investigations of Triplex steels using high resolution characterisation methods should give a more detailed insight into the role of κ -Al(Fe,Mn)₃C carbides on the deformation behaviour. Our results have shown that it is particularly pertinent to focus on alloys containing high Al concentrations (>8 wt.%), as they exhibit a low density and offer the possibility of covering a wide and well tunable range of mechanical properties via ageing (scalability): YS, 500–940 MPa; UTS, 710–1020 MPa; TE, 78–8%.
3. Following our RAP approach the 45 different material conditions were synthesized, processed and tested in 35 h by employing modified and comprehensively adapted casting, hot rolling and sample preparation techniques. This represents a time advantage of a factor of 6–10 compared with conventional metallurgical synthesis. As it allows fast screening of the mechanical properties dependent on chemical and thermo-mechanical parameters, RAP has significant potential as a novel tool for efficient alloy design, ideally employed in interactive cooperation with simulation and high resolution characterisation methods.
4. A direct comparison with results obtained for conventionally synthesised materials reveals that the quality of both the mechanical data and the obtained chemical composition precision of the RAP synthesized alloys was slightly reduced (typically below 10%). This minor decrease was expected in view of the applied multiple casting operations and the geometry of the tensile samples (e.g. surface effects). However, we observe that the data quality obtained by the RAP approach was sufficient to spot relative trends in the bulk material behaviour. The absolute values obtained were in reasonable agreement with the published data, allowing reliable pre-selection of material parameters for a more detailed analysis.
5. The applied synthesis route, via casting and hot rolling, of the RAP approach is best suited to the investigation of the most common structural materials, such as steels, nickel, titanium or aluminium alloys. Future developments aim at a further increase in the processing speed and applicability for different metallic systems, e.g. by

the implementation of alternative synthesis processes, such as strip casting and powder metallurgy techniques, respectively. The modular concept of RAP facilitates the replacement of single processing steps (e.g. synthesis) and expansion of the employed testing procedures (e.g. fracture toughness) according to specific characterisation needs.

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