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Rapid Alloy Prototyping for a range of strip related advanced steel grades

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Abstract

Over many decades, the traditional route for material product developments, especially in the steel industry has been the laboratory VIM cast route at scale of 25 to 60kg, followed by through-processing of steel ingots involving hot rolling and cooling as well as further downstream processes to simulate finished cold annealed rolled and coated products. This traditional route has so far delivered value for optimising current grades and process routes as well as developing new products prior to production implementation. However, in order to accelerate process and grade developments even smaller scale and faster laboratory synthesis and processing is desired. The AccMet project [1] developed strategies for new alloy development [2,3] and this needs to be further developed to account for the complex processing route for strip steel production. Strategies combining small scale laboratory alloy processing routes, together with mechanical/thermal testing and modelling are being developed, ranging from 20-30g to 4.5 kg [4-8].

This paper summarises current Rapid Alloy Prototyping (RAP) approaches and rationale developed under a new UK Engineering and Physical Sciences Research Council (EPSRC) Prosperity project between Tata Steel and the Universities of Swansea and Warwick (WMG). Specific attention is paid to the overall experimental methodology as well as benefits (throughput) of small-scale manufacturing and testing, the generation of representative microstructures for a range of strip grades as well as ways of integrating new concepts which bridge the physical length scale. A range of experimental facilities (20-40g) based on a powder route and induction melting (IM)/heat treatments is being developed to provide material for hot/cold rolling/annealing prior to mechanical testing. Modelling and testing to account for mechanical test specimen size effects for small scale RAP samples is being carried out to ensure consistent mechanical properties are obtained. This small-scale RAP is also being complemented with an intermediate material route operating between 200g and 4.5kg using centrifugal casting and small size ingot vacuum induction melting respectively to provide additional material and throughput sitting alongside the more traditional pilot-scale 25-30kg route. Finally, the 25-30kg standard route is being reviewed to provide a bridge to the laboratory routes through various innovative concepts. This paper concludes with a review of future activities and challenges for effective development and implementation of a range of small scale experimental and pilot manufacturing lines.

Keywords: Rapid prototyping, advanced metallurgy, size effect, Alloy optimisation and development, DP steels, data modelling

1. Introduction

The Steel Industry needs transformation to accelerate development of added value products whilst increasing efficiency, financial return and sustainability. Conventional optimization and development of current and new steel grades is slow and iterative and therefore requires a new approach. Although combinatorial approaches to developing new alloys (product and coating) are not new (see [1-3,7-8]), their application in the UK and steel industry is. A new 5-year project

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under the Engineering and Physical Sciences Research Council (EPSRC EP/P020755/01) Prosperity Partnerships has been established to : 1/ Create and implement a new steel innovation cycle, binding industry and academia together, 2/ Accelerate the development of next-generation steel products by at least a factor of 5, 3/ Allow a wider-ranging and imaginative approach compared to conventional methodologies through a de-risking strategy. This project links through EPSRC, the leading UK institutes (Universities of Warwick and Swansea) and members in steel innovation and development. Integration and optimization of both academic and industrial research coordinated by Tata Steel should promote an effective way in harvesting and streamlining development, application and implementation, giving both blue sky and high risk innovative research the ability to be tested and supported by industry in a closed-loop manner. During its first year of development, the project has seen four RAP lines being established to cover aspects of physical length scales as well as the development of a new Intrap insert methodology to bridge the physical length scale between the small 20g route and 30kg route. An initial optimisation using RAP 4.5kg of microstructure and process parameters on DP600 and DP800 steel grades has also been carried out and is briefly presented here. New innovative developments in Machine Learning (ML) and data analytic algorithms ranging from self-organizing maps, clustering techniques to neural networks and applied to selected benchmarking and production grades, providing combinatorial process and product capabilities have also been developed. Additionally FEM models are being developed and applied to study a range of mechanical tests and size effects covering tensile testing, small punch and shear compression with finally the development of a through process physical metallurgical model initially targeting Continuous Annealing lines (CAPL) to study the influence of intercritical annealing, recrystallisation, processing and initial phase fraction on final microstructure properties. These last two strands of these project activities are not included in this paper which mainly focuses on the experimental establishment of RAP lines.

2. Methodology

2.1 RAP 20-40g [University of Swansea]

This route is primarily a powder route for current/new alloys as well as a solid route based on production alloy remelting. As shown in Fig.1, this process route integrates powder selection and weighing, compaction to reduce porosity prior to induction melting (IM) using various crucibles to generate typical mini cylindrical specimens of max 12 dia x 30mm. The main difference between 20 and 40g is the ability of using the 40g route to have more reproducibility over melting/composition analysis (within 0.02 wt% target), to also have more material in one melt to cover a wider range of processing conditions as well as enabling composition analysis via OES for instance. On a 20g, usually three samples (melts) are required to cover one composition for validation, therefore decreasing by a factor x3 the throughput as compared to 40g. A 40g single melt can also offer advantages in terms of end shapes as well as producing 2 to 3 small (ASTM25) and more mini non-standard tensile specimens (Fig.2). Initial length is typically 10x10x70mm versus 10x10x35mm for a 20g RAP rectangular specimen (Fig.3). Following small ingot melting, composition and microstructure characterisation is carried out using OES, SEM/EDX and XRF (for segregation). Fig.4 shows currently typical XRF Mn segregation obtained along the length of the mini 40g ingot. Homogenisation treatments can then be imposed to homogenise the as-cast structure prior to rolling (currently cold) and final heat treatment to simulate CAPL annealing/quench and tempering as well as assessing final structure-properties (tensile, shear, hole expansion HE, etc.) Other thermal-mechanical simulations can also be carried out by feeding RAP 20/40g samples into Gleeble uniaxial, plane strain compression (PSC) or dilatometer to break down the as-cast structure and impose different thermal-mechanical controlled rolled (TMCR) schedules as well as deriving CCT/TTT data. So far, using this route, a range of benchmark alloys (with varied C, Mn, Si addition) has been manufactured to cover DP grades, IF steels, CMn structural as well as low C HSLA. This route is being further developed and used to manufacture IM alloy inserts for the standard 30kg laboratory pilot route giving capability to higher range of deformation sequence. Key at this scale is reproducibility of composition (so far with 0.02wt% even at RAP20g), ability to add and control several alloying elements, matching final length/width of 20/40g RAP specimens to usable mechanical tests as well as giving appropriate processing conditions (strain, strain rate, temperature, etc.)

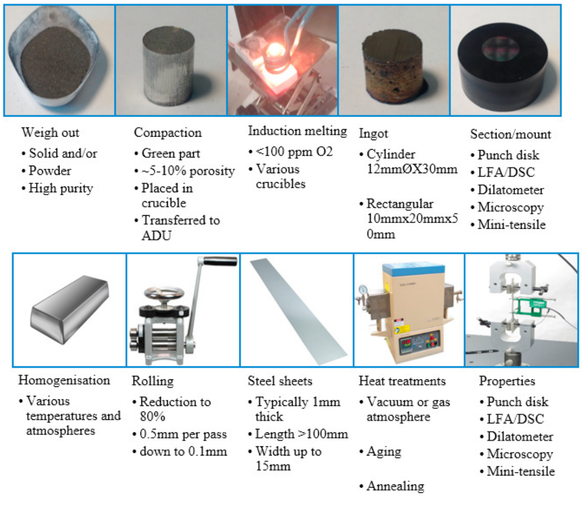

Fig. 1: 20/40g RAP through process route.

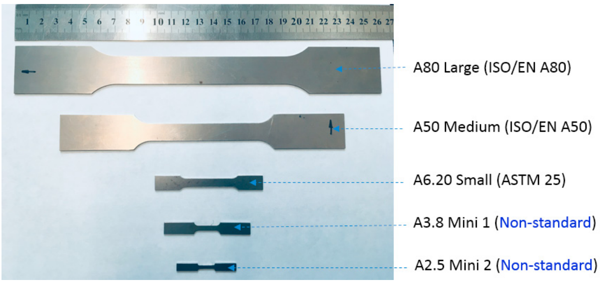

Fig. 2. Range of tensile specimens.

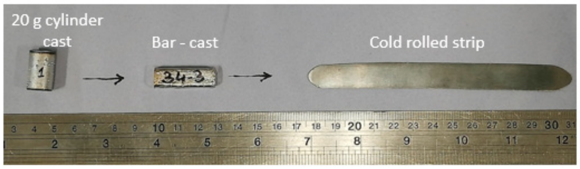

Fig. 3. Example of 20g RAP specimen cold rolled to 1mm strip.

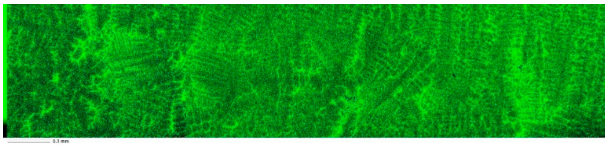

Fig. 4. EDX map for Mn segregation in DP RAP 40g product.

This route due to its length scale, will not be able to absolutely match a conventional Hot Strip Mill route (HSM), also not a finishing schedule (usually 32-38mm down to 1.5mm) as well as a 30-60kg standard VIM laboratory route. Solidification / cooling also requires further optimisation as to develop characteristic secondary dendrite arm spacing, grain size and segregation. De-oxygenation (< 100ppm O_2) also requires studying to approximate production or VIM cast laboratory material. This route can also be used in remelting production material with/without addition of supplementary alloying elements for either producing material for design of experiment (DoE), for instance studying processing and ingoing phase balance of DP steels during CAPL simulation stage or tweaking material composition. Not all the benefits can arise from running a 20/40g RAP line to the end product (i.e. a tensile test or punch test), but many benefits can be realised earlier or through the process, for instance through the assessment of loads/torque/power from derivation of mean flow stress function of strain, strain rate, microstructure function of alloy composition, to also looking at influence of residuals/scrap addition onto for instance ductility and/or oxidation issues (e.g. influence of Cu/Sn/Ni, etc.). For the 40g, excluding detailed characterisation, it is anticipated that a throughput of 50 to 60 alloys per week could be achieved. A standard laboratory 30kg VIM pilot route manned by two operators involved in casting, rolling and subsequent heat treatment can take up to 9 weeks for 1 alloy to be processed and 40-45 weeks for a DoE for a CAPL simulation. Whilst large quantity of material can be obtained from a 30 or 60kg route, it is often too narrow to test transverse properties using standard ASTM specimens. When comparing a normal standard 30kg or 60kg route in a pilot environment, the small scale route can represent a speed up of at least a factor x20 (for same number of specimens) on the assumption that finished specimen RAP 20/40g sizes can be consistently used by either standard or non-standard mechanical testing (see Fig. 2). On the cautious side, a RAP 20/40g will require efficient and speedy characterisation, machining and quality insurance (QA) to realise the benefits claimed above, ideally using in-situ facilities and additional operators. Consistency and uniformity of properties through tweaking/optimising solidification / heat treatment profiles are also key aspects to master. Performing commissioning of these small-scale RAP lines together with understanding of capabilities versus production operating processing windows of selected grades mostly when dealing with TMCR, non-recrystallisation temperature (Tnr), cooling rate to simulate ROT is also critical. Similar to the 30-60kg traditional laboratory route which is a standard route for many steel producers for product development, developing a critical knowledge of all +s and –s of these small RAP lines will take time and effort, but is needed. On the capital/cost front, a small 20/40g line is very cost effective (at least by a factor x10), requires less services and footprint allowing also parallel processing to be implemented for further increases of throughput. Operational (process timing) and safety measures need also to be optimised in view of the processing physical scale and powder route. Implementation of robotics and process control at such a small scale remains a challenge to be addressed.

2.2 RAP 200g [University of Swansea]

This process is under development based on a solid or pellet alloy material route, induction melting and centrifugal casting in argon to produce a range of larger ingots of up to 200g in a variety of shapes up to 70mm length (and width) and 10mm thick (typically 10x50x70mm). A new mini hot mill with heated cassette/rolls (max 250^{o}C) is being purchased to roll these ingots to impart a reduction of up to 80% whilst minimising heat losses in the roll gap. Steel sheets of up to 300mm length should be able to be manufactured in a range of widths to accommodate various tensile test designs inc. the ASTM A80 standard which is the de-facto standard for release production material (see Fig.2). Additional processes such as the simulation of a Run Out Table (ROT) are being looked at. Fig.5 shows a typical process manufacturing route for the RAP200g where the main differentiated factors are the addition of a centrifugal caster as well as a mini hot mill. Similar to the 20/40g RAP line, thermo-mechanical simulators (Gleeble, Dilatometer, etc.) can be added to provide thermal-mechanical-microstructural data as well as also providing a mean to further shorten the process. The horizontal centrifugal casting is in principle ideal for producing high quality and fine grain structure through the high speed mould rotation exerting high G-force on to the cold mould (function of v2/r where v is the linear metal velocity and r the radial distance). This facility is being commissioned at the time of writing this paper and will need to be optimised regarding metal pouring, but also assessing whether aspects of surface turbulence are present, together with assessing the liquid shape (most likely parabolic). Detailed characterisation using XRF, SEM/EDX will need to be done in relation to impurities, oxides with high drag and low stokes velocity. The benefit of this intermediate scale facility is the balance between throughput and physical length scale allowing cast feedstock to be hot rolled in a mini mill with then the ability to incorporate ROT simulation as well as downstream processes. Again, the number of finished parts should be increased according to weight increase (X5) with only one additional process added.

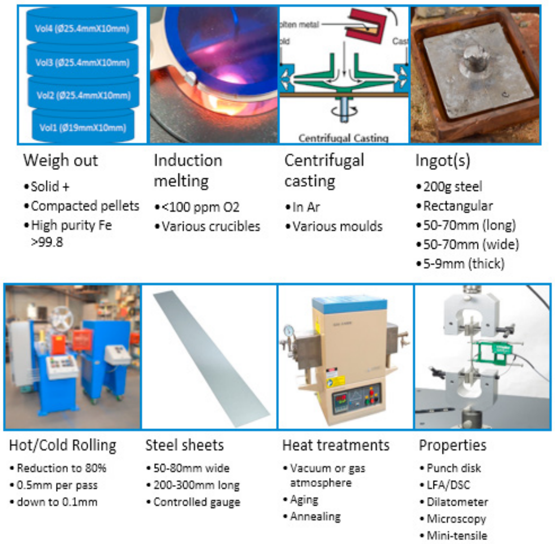

Fig. 5. RAP 200g as developed at the University of Swansea.

2.3 RAP 4.5kg – max 10kg [University of Warwick]

Further scaling-up the weight of the ingot is a route being developed at Warwick University within the ASRC (Advanced Steel Research Centre) steel laboratory of WMG (Warwick Manufacturing Group). This route is still considered as a RAP experimental line as being capable of developing and processing a composition within two weeks including machining of tensile samples, full compositional and microstructural analysis. It consists of more traditional and standard laboratory processing steps such as a small VIM (max 10kg), a hot and cold mill, a range of furnaces as well as an annealing furnace combined with a fluidized bed for continuous annealing simulation as well as imparting homogeneous cooling and improved surface state. Fig. 6 shows the through process manufacturing stages implemented at WMG-ASRC.

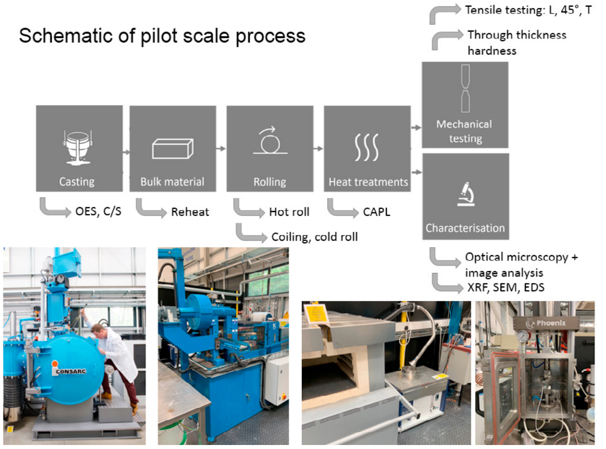

Fig. 6. WMG medium laboratory pilot scale for rapid prototyping (4.5 to 10kg).

The benefits of this physical length scale is the ability to tune the casting size to the secondary dendrite arm spacing (SDAS) function of cooling rate and ingot shape. The use of a small VIM unit allows a well-controlled composition to be made with either Al killed or C boil route. Typical ingots so far produced and optimised for the study of DP 600 and 800 grades are 30x80x220mm, giving 3 or 4 initial “transfer bar” as-cast feedstock feeding the Hille25 small roll diameter/bite angle hot mill. Recently the mill was upgraded to enlarge the initial collar gap. More precisely, this RAP line can deliver ASTM E8 sub-size specimens following CAPL simulation, with then, when sub-size tensile specimens are optimised, able to deliver more mechanical property statistics. The various processing steps including critical steps for characterisation and machining are shown below in Fig.7. Work is ongoing to study the balance between hot rolling (HR) and cold rolling (CR) reduction, especially for DP grades, but general practices are that ingots of ~30mm thickness are hot rolled to 3/2.5mm, slow cooled in a coiling furnace before being given a 45 to 70% cold rolling reduction. The balance between HR and CR reduction is important for CAPL simulation and final properties (strength v ductility). Again, even at that operating physical scale, the overall reduction sequence is lower than the one imposed on a standard 30kg laboratory pilot line, which depending on installation can start with ingots of 70 to 100mm^2 cross section followed by two stages of rolling: cogging to transfer bars (32-40mm) and then finishing rolling to 2-3mm. As a direct comparison, this RAP line takes roughly same feedstock as rolled transfer bar production material prior to the finishing mill but in as-cast conditions. In Section 3, some results are shown regarding optimisation of DP microstructure and properties using the RAP 4.5kg, by optimising secondary arm dendrite spacing, reduction and heat treatments.

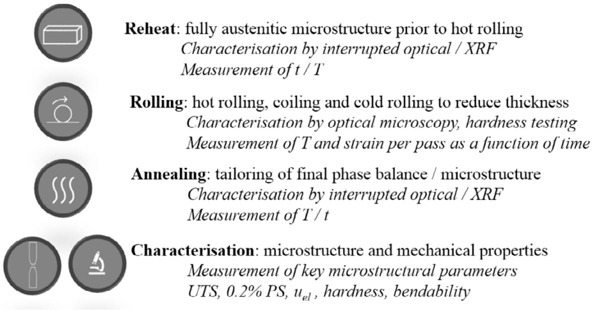

Fig. 7. WMG 4.5kg RAP sequence of process steps.

2.4 Intrap insert methods on 30kg Laboratory route [University of Swansea, Tata Steel].

The laboratory pilot route (30-60kg) for product development and optimisation is still the de-facto standard for most steel producers but as stated in the background to this project, even when manned by more than two operators, the through manufacturing process is slow and cannot cope nowadays with aspirations of steel producers to develop new and improved grades in usually a 6-month lead time. Through process elapsed time from large VIM melting to large simulation of annealing cycles using a range of processing conditions (soaking temperature, over ageing, cold rolling reduction, coiling temperature, etc.) and varied alloy composition lead to extremely large design of experiments well (even on a fractional design) in excess of equipment availability. As an example, adopting a full factorial 3-level design of experiment for studying a CAPL simulation based on 5 temperatures and a line speed will require 3^6 or 729 experiments for one steel composition. This experiment is for illustration purpose only and assumes that no or minimum physical / numerical simulation is available. A typical annealing simulator (CASIM) annual availability is roughly ~1000 experiments, therefore if one is to carry out such an experiment, the equipment and operators will be tied-up for a full year on one composition for one steel grade. Usually steel producers maintain a new product strategy of more than 10 new grades across all sectors for development, highlighting the clear bottlenecks of the traditional method and need to supplement this strategy with more appropriate rapid prototyping as well as modelling and AI tools. Therefore, considering the benefits of larger scale experiments for better mimicking production processes in terms of reduction and heat transfer, a new route bridging the physical length scale has been developed based on RAP20/40g IM material inserts (see Section 2.1) implanted into transfer bar blocks from either the 30kg laboratory scale or production routes. Fig. 8 shows the InTrap methodology based on RAP20/40g IM inserts which for this initial validation exercise, used DP800 remelted products. A DP800 grade 12x15mm cylinders were initially inserted though the thickness of a 15mm HSLA transfer bar to prove the concept of reheating, HR and CR deformed sheet/insert prior to annealing and tensile testing. Fig.9 shows a respective microstructure as well as size limitation of insert deformed material versus tensile test specimen based on initial insert dimension. Most of the insert material which ends up ellipsoidal nearly fitted the small ASTM25 tensile specimen selected, however this process is being further optimised to meet tensile dimensions as well as ensuring that recipient parent production material is in close match to the insert composition to avoid decarburisation as well as decohesion. However, overall this process has opened the possibility to link the small 20/40g RAP route to the larger and standard laboratory route. IM samples can be inserted in various shapes/forms as well as state of deformation if for instance deformed in a Gleeble simulator prior to insertion. The overall manufacturing process is relatively efficient, mostly for through thickness insertion and allows also, depending on transfer bar parent material, study of the influence of localised deformation based on shape/volume and location of insert. Many inserts can be “implanted” into a transfer bar or initial cast VIM ingot increasing throughput and relevance to the real production process. This process at larger scale can be applied also to production slab material. Surface state evolution such as friction can then be also studied.

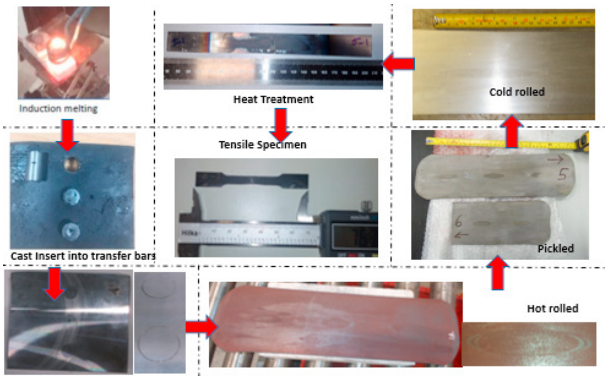

Fig. 8. InTrap IM RAP 20/40g insert methodology.

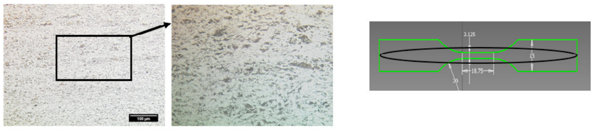

Fig. 9. First InTrap DP material inserted into an HSLA showing insert final deformed/annealed shape versus ASTM25 tensile specimen.

Developments in Intrap material methodology are now focusing on optimised shape/type from either RAP20, 40g and potentially 200g into transfer bar material as shown in Fig.10.

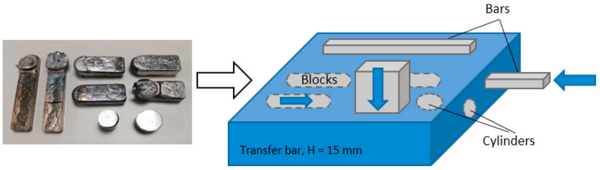

Fig. 10. Optimisation of Intrap methodology.

Fig.11 shows reconstructed 2D microstructures at three key stages of the Intrap material, HR, CR and post CAPL simulation. Further optimisation is still required to minimise compositional changes by optimising the parent material and processing conditions, but overall this initial exercise proved that the concept has potential in delivering added-value.

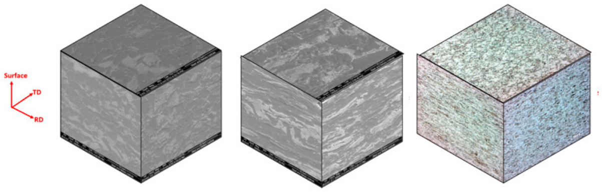

Fig. 11. 3D representation of 2D microstructure after HR, CR and annealing (DP simulated grade).

3. Example of initial optimisation of DP600-800 using RAP 4.5kg *[University of Warwick]*

Effort has been focusing on optimising the RAP 4.5kg at WMG to produce representative microstructure and properties (YS,TS, El%) in line with production DP 600 and 800 material. The benchmarking and optimisation philosophy is presented in Fig.12 by optimising the secondary dendrite arm spacing λ_2 (SDAS) according to Volkona [9] as well as reduction between

HR and CR, to produce a microstructure with similar band spacing, grain size and phase fraction. This was done by imposing a CAPL cycle in line with plant data and distribution as well as meeting initial DP composition from the VIM process. This detailed commissioning was done initially on DP600 with a target thickness of 1mm, grain size of ~6µm, band spacing of 10µm and finally a second phase fraction of ~23%. Fig. 13 shows current mechanical tensile property results obtained by simulating DP600 through the entire VIM melting to exit CAPL. New rules have been developed to predict band spacing as a function of overall reduction, together with phase fraction as a function of autotempering time. The RAP 4.5kg allows for testing tensile properties at ASTM E8 subsize specimens but should be able to benefit from the current study of size effect in tensile specimens when this work is completed further providing statistics as well as possibility to test transverse properties.

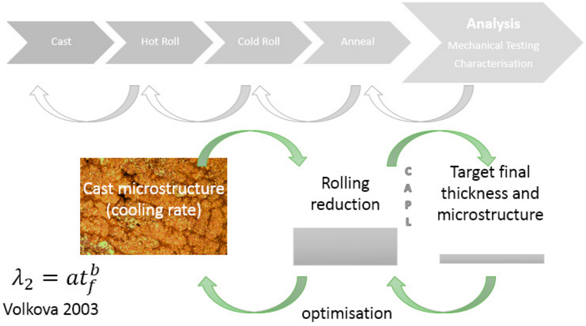

Fig. 12. Optimisation strategy for DP grades – RAP 4.5kg.

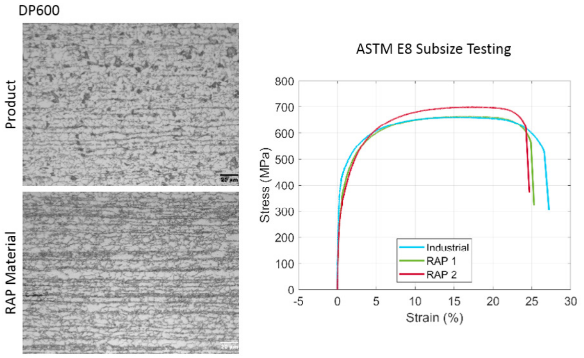

Fig. 13. DP 600 RAP4.5kg tensile mechanical property post CAPL.

A similar exercice (not shown) has also been done for DP800 with very encouraging results in terms of band spacing, phase fraction and final mechanical properties. Work is currently focusing on understanding effect of microsegregation (Mn) onto properties as well as benchmarking other grades.

4. Conclusions and Way Forward

This paper summarises initial developments and current status for the establishment of four Rapid Alloy Prototyping lines operating in the UK at three different physical length scales, together with a new methodology to bridge length scale allowing small Induction Melted (IM) RAP 20g inserts (InTrap method) to be embedded and processed into the standard 30kg laboratory route. Good progress has been made to start commissioning and optimising these lines targeting DP steel grades from melting to annealing with already representative mechanical properties and microstructure as compared to production. Work is now focusing on processing a range of benchmark grades (IF, structural, HSLA, etc.) and establishing a robust process supported by underlying physical metallurgy, modelling, DoE and data science. The expected throughput should be at least ten times faster, depending on lines or combination of RAP lines. Work is also focusing on meeting alloy composition consistency and uniformity (mostly when operating at small scale 20/40g) and mapping envelope of RAP processing conditions versus plant process/product distributions. Further work is also required to assess combined effect of process steps on changes of microstructure and how discrete processes compare with the full through process. It is also of interest to see what additional benefits can be obtained to further speed-up the RAP process using a thermal-mechanical simulator such as Gleeble. Operating conditions/standards for RAP lines supported by characterization standards/references will also be very important to be established. Finally, the RAP lines offer possibilities to study surface state function of alloying using thermogravimetric analysis (TGA) and deformation. Advantages and drawbacks in relation to for instance throughput, together with any future improvements have been highlighted in this paper. This multi-strand activity should then contribute to the establishment of a combinatorial, smart manufacturing process for advanced and rapid alloy and coating developments.

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