A Review on Steel Thixoforming

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Abstract : Steel is a particularly challenging material to semi-solid process because of the high temperatures involved and the potential for surface oxidation. Here the experience of semi-solid processing will be reviewed and the current situation in relation to commercial application assessed. The review will include discussion of the range of potential steel materials which are amenable to thixoformingn and identification of suitable steels; modelling of die fill and rheological properties of semi solid steel; technology considerations for industrialisation; die development and the properties of thixoformed products.

Keywords: Steel; Thixoforming; semi-solid; industrialisation

1 Introduction

For successful thixoforming [1], the microstructure in the semi-solid state must consist of spheroids of solid in a liquid matrix. The material then behaves *thixotropically* i.e. when it is sheared it flows but when allowed to stand it thickens again. This microstructure may not be necessarily be present in the raw material but could be obtained after re-heating. The thixoforming process is baptized *Thixoforging* when the solid fraction is high and *thixocasting* when the solid fraction is low. The Flemings group experimented at the pilot plant scale with *rheocasting* (i.e. forming in the semi-solid state direct from the cooling liquid metal rather than having an intermediate solidification step). They formed a series of steels including AISI 304 and 440C [2] but found that oxide inclusions tended to lead to variable mechanical properties. Aluminium casting alloys and magnesium alloys proved highly amenable to the process and in the 1980s there was extensive effort on the industrialisation of the thixo-processes for light alloys, with the more challenging high temperature alloys moving into the background.

In the early 1990s, a group based in Sheffield, UK, led by Kirkwood, Sellars and Kapranos, revived interest with a consortium of UK based companies including Rolls Royce. The Sheffield equipment used a vertically upwards acting ram within a vacuum chamber (to avoid oxidation of the slug during heating) and a graphite die. Such a die would not be suitable for long production runs but was relatively cheap in comparison with tool steel dies and therefore suitable for an experimental programme. The Sheffield group saw the potential of ceramic dies for thixoforming high temperature alloys and patented this idea [3]. The Sheffield work compared results from spray formed materials [4] (a relatively expensive process) with the RAP (Recrystallisation and Partial Melting) process [5] which simply involved material in the conventionally cast and heavily deformed state for T15, M2 and H13 tool steels and 440C stainless steel, with encouraging results. The consortium then focussed on M2 tool steel for extensive trials [6, 7], partly because it has a reasonably large thixoforming window (~150˚C) in comparison with stainless alloys, and partly because its high alloy content leads to a relatively low solidus.

At the end of this consortium project in the early 1990s the main difficulty was that there was no commercial manufacturer of equipment for steel thixoforming and companies were not willing to take the risk on the developmental stage.

In parallel with the Sheffield work, the Thixoforming IBF in Germany led by Kopp and Hirt was also aiming to thixoform steels. It became clear that without the facility to shroud the billet during heating, the build-up of oxide on the surface prohibited successful processing. A major collaborative project on thixoforming was initiated at RWTH Aachen and issues such as die longevity started to be explored. Work at Aachen [8] showed that, when thixoforming M2, the stresses induced in the die tool steel were several times the yield strength and therefore ceramic inserts were necessary. Meyer and Bleck (also at Aachen) [9] showed Differential Thermal Analysis (DTA) results for M2 and compared with the calculated results from a thermodynamic prediction package, techniques which become powerful in enabling amenable steels to be identified and characterised. At a 1997 conference, work was also presented highlighting that commercial wrought high speed steel bar could be thixoformed without any special treatment [10]. As we have progressed into the 2000s, major EU consortia (‘THIXOCOMP’, ‘Adapted Steel Parts’ and COST 541 ‘THIXOSTEEL’ and SFB289 have aimed to overcome the major barriers to exploitation).

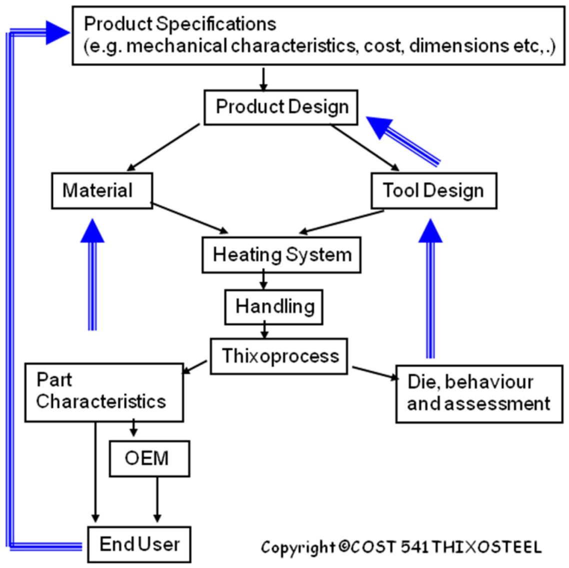

Fig. 1. Thixoforming of steels, conception and realization diagram

The themes in this review paper are inspired by the above mentioned consortia achievements which are mainly routes to thixoformable starting materials and identification of suitable steels; modelling of die fill and rheological properties of semi solid steel; technology considerations for industrialisation; die development and the properties of thixoformed products. The link between the different themes is summarised in **Fig. 1**. Some concluding remarks will then be given.

2 Routes to thixoformable steels and identification of suitable grades

As identified above [2], the Flemings group used rheocasting to obtain thixotropic material. However, this requires the facilities to melt steel and hold it in the liquid state; many university laboratories have not had facilities on this scale available. Rheo-routes are preferable as there is an energy saving relative to the routes where there is an intermediate solidification step; this saving is particularly crucial for a high melting temperature such as steel. Routes where molten steel has been treated include the following. A group based in Seoul has produced stainless steel billets by Magneto Hydrodynamic (MHD) stirring and shown excellent microstructures [11]. The University of Science and Technology in Beijing has also electromagnetically stirred and then rheorolled spring and stainless steels [12]. Strip obtained showed good mechanical properties. At CRM in Liege in Belgium they have developed a specially designed hollow jet nozzle for continuous casting which allows powder injection and a low superheat to be combined to obtain a globular structure in the core of a continuously cast steel billet [13].

Various workers [14-17] are investigating the use of a cooling slope (similar to the New Rheocasting-NRC-process or the work of Haga and co-workers [18, 19]) with some success. Vibratory casting has also been used [20], including to obtain functionally graded material based on hydroxyapatite and 316L stainless steel [21].

There has been interest in whether steel can be thixoformed in the standard state in which it is received from a steel supplier. Bulte and Bleck [22] compared the microstructure after quenching from the semisolid state for 100Cr6 rolled bar, laboratory cast billet and laboratory cast billet with liquid core reduction. They found no significant difference and therefore suggest primary as-cast billet can be used. Omar and co-workers [23, 24] have demonstrated that the high performance steel HP9/4/30, which is very difficult to form by other routes, can be successfully thixoformed in the as-supplied state. Note that in the as-supplied state the material is already recrystallised and therefore this route does not fit with the ‘classical’ solid state routes of RAP and SIMA (Strain Induced Melt Activated).

The original approach to identifying suitable steels involved examining the phase diagram. This showed that low alloy, low carbon steels had narrow solidus-liquidus ranges and therefore process control was likely to be difficult. Work therefore focussed on high alloy, high carbon compositions, which also tend to be the compositions which are the most difficult to work by conventional means.

As mentioned in the Introduction, the approach of combining experimental results from Differential Thermal Analysis (DTA) alongside thermodynamic prediction was then developed.

Kazakov [25] identified parameters for the liquid fraction versus temperature curve for alloys to be suitable for thixoforming. These have been adapted for steels [26]:

- The solidus and liquidus temperatures, T_S and T_L, which must be as low as possible;
- The temperature at 50% liquid, $T_{50\%}$, which must be as low as possible;
- The melting interval, T_L-T_S, which must be as large as possible;
- The semi-melting interval, $T_{50\%}$-T_S, which is of interest as it approximates to the thixoforming window;
- The slope at 10 and 50% liquid, $(df/dT)_{10\%}$ and $(df/dT)_{50\%}$, which must be as low as possible to ensure a small sensitivity of liquid fraction to temperature.

Figure 2 shows the measurement curves for a conventional C38 steel grade and a steel where the composition has been modified specifically for thixoforming [27].

The modified alloys have a lower soliduus temperature and a wider process temperature range [28]. However, the effect on resulting mechanical properties must also be carefully weighed.

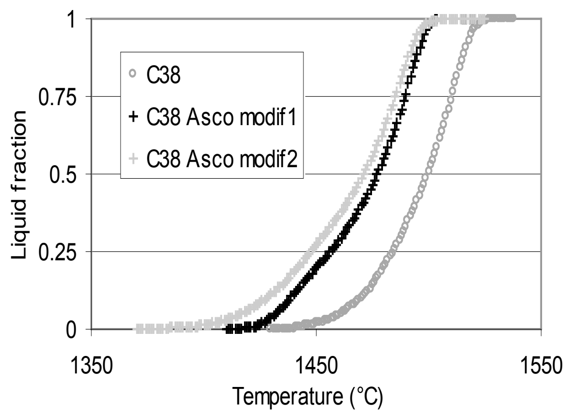

Fig. 2. Liquid fraction behavior vs. temperature, comparison between classical C38 steel grade and modified one.

Puttgen and Bleck [29] have carried out extensive DTA experiments, showing that the cold work tool steel X210CrW12 and the tool steel HS 6-5-3 have a wide semi-solid area (due to the dissolution of different carbides) but a range of other steels have much narrower semi-solid regimes. The bearing steel 100Cr6 is also highly suitable. This steel and X210CrW12 have therefore been extensively investigated in terms of the phase formation during processing [30]. Figure 3 summarises liquid fraction behaviours of the investigated steels. Hallstedt et al. [31] have also used thermodynamic prediction, suggesting that important criteria are the slope at 50% liquid and the temperature ranges between 40% liquid and 60% for thixocasting and 20% and 40% for thixoforging.

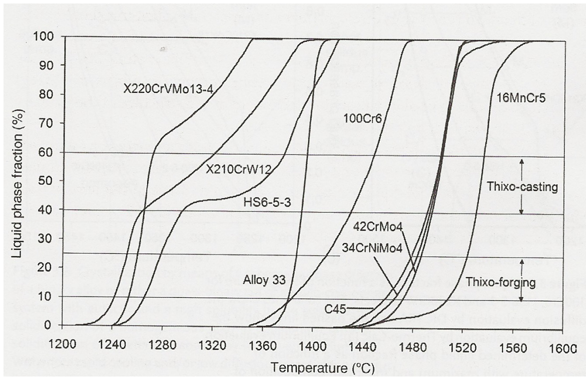

Fig.3. Liquid fraction behavior vs. temperature for different steels using DTA at a heating rate of 10°C/min [29]

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Li et al. [32] have identified how the inverse peritectic reaction can lead to spheroidisation in stainless steels in the semi-solid state. They argue that spheroidisation will always occur in an alloy with a peritectic reaction independent of the morphology in the solid state. They have named this phenomenon Inverse Peritectic Induced Spheroidisation (IPIS).

3 Modelling of Die Fill and Rheological Property of Semi-Solid Steels.

Modelling of semi-solid processing has been reviewed by Atkinson [33, 34]. The high temperatures involved with steels make the experiments particularly challenging. Modigell et al. [35] have developed a high temperature Couette rheometer to analyse flow properties up to 1600 ℃ and have characterised X210CrW12. Cezard et al. [36] have derived input parameters for their finite element simulation using Forge 2 software from successive comparisons of the simulated load-displacement curves with experimental ones for compression tests on steel semi-solid at different temperatures. The constitutive equation is based on the micro-macro approach. Omar et al. [37] have also obtained viscosity-shear rate relationships from load-displacement curves. Hot compression is used to obtain flow stress curves but only around the solidus temperature [38]. Shimahara et al. [39] have designed a special cylinder sample with a shell to avoid the problem of collapse of the sample during hot compression as liquid develops. At temperatures just below and just above the solidus, Solek et al. [40] and Kang et al. [41] have carried out experiments with a Gleeble simulator to characterise rheological properties.

At present modelling of die fill is not well developed because of the difficulties with obtaining experimental parameters but the principles should be similar to those for modelling semi-solid die fill in general [33]. However, modeling can still be very helpful with identifying where defects are likely occurring and adjustments which should then be made to the thixoforging parameters. Figure 4 shows such an example.

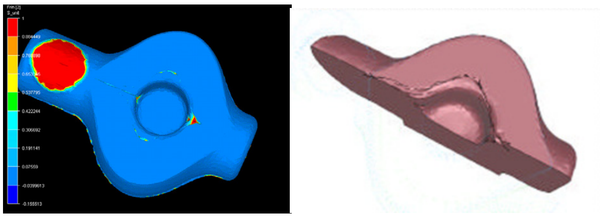

Fig. 4. Material flow modelling (left) and real part (right) showing defects around the central air intake hole. The modelling led to adjustments in the process and successful component production [42].

4 Technology Considerations for Industrialisation

The industrial development of steel thixoforming must be accompanied by knowledge and control of the various parameters for the process: the identification of the steel grades; the homogeneous high-temperature heating of the slug before deformation; conception or re-conception of the part to adapt it to the process; the parameters for forming including ram velocity profile, hold time and pressure at the end of the stroke, die temperature, die material. The handling system is also important; it allows the slug to be transferred, in the semi-solid state, between the heating zone and the tooling and to evacuate the thixoformed part so as to ensure the quality heat treatment of the part. All these aspects contribute to achieving a cost for the finished part which is competitive with more conventional processes [43].

Industrial developments include: non-contact temperature measurement during heating [44]; the use of fuzzy logic to control the heating [45]; optimising the induction coil geometry [46]; prediction of thermal losses during transport and after insertion into the die [47]; a device on the hydraulic press to counteract the decrease of the punch speed in the final stages of the stroke [48, 49]; automated handling and industrial vision systems [28, 50, 51].

4.1 Induction heating

The slug induction heating is one of the essential operations of the thixoforming process [52]; the temperature distribution through the slug just before forming must be as homogeneous as possible to enable a uniform distribution of liquid fraction through the slug volume. In addition, the heating schedule must be such that, whilst achieving the above requirements the time to temperature is as short as possible. The heating operation regulates the process productivity. To ensure a comparable productivity to other hot forming processes, with induction heating it is necessary to heat slugs on a carousel. During the induction heating operation, the slug will be protected from oxidation by an inert gas atmosphere e.g. argon.

4.2 Transfer and handling systems

Robots are necessary to achieve reproducibility of transfer and handling operations. A first robot has to locate the slug in the heating zone and then transfer the semi-solid slug to the forming zone. A second robot transfers the part towards the evacuation or heat treatment zones. This second robot can also clean the tool active zone and carry out lubrication of the tool. Alternative handling systems give also good process reliability but are less accurate than robots.

4.3 Press and forming equipment

A thixoforming press must be able to be operated under either load or displacement control. The press will be either hydraulic or a screw mechanical press. For operation in the semi-solid state, the movements must be fast and as much as possible constant on the main axis to obtain high speed deformation of the semi-solid metal in tools. A hydraulic press presents some advantages with the possibility to use some slides or pistons and with a very large surface table.

4.5 Control of manufacturing

Quality control includes inspection of: dimensions, geometry, visual defects, magnetic-particle inspection, metallographic structure and hardness. The parameters must be adapted to customer specifications but also interact with the process e.g. dimensions affect re-heating and hence reproducibility.

5 Tools and forming dies

Thixoforming of steels presents a particularly severe requirement on die properties. The process is characterised by high temperatures, temperature gradients, pressures and considerable stresses. This process requires materials with high-temperature corrosion and thermal shock resistance under load to meet performance and durability requirements. Forming semi-solid steel requires special dies which must be able to withstand repeated contact from material forming at temperatures of around 1250°C. Thixoforming steel dies should have high temperature mechanical strength, wear, fatigue, creep, shock, corrosion and oxidation resistance properties. One of the greatest challenges in thixoforming of steel is developing a die to meet these requirements. Recent research has investigated a wide range of high temperature coatings, bulk ceramics and surface laser processing techniques in order to prolong the life of the die during steel thixoforming. Cycles producing hundreds of parts have been achieved in practice using ceramic die materials. For the industrialisation of the process, dies for thixoforming of steel alloys need to be capable of producing closer to a hundred thousand or more production cycles. A series of different potential solutions have been investigated including: oxide PVD coatings on TZM (a Mo-based alloy) [53]; PACVD alumina films on hot working steel [54]; ceramic inserts (Si_3N_4, $ZrSiO_4$) in a hot working steel shrink ring [55].

In the SFB289 project [56], three types of materials have been compared: thin film PVD and PACVD; thermally sprayed thick coatings and bulk ceramic materials. The results showed that the behaviour was sensitive to the stresses at the surface of the die and therefore the die should be made of different materials in different positions.

Gas pressure sintered Si_3N_4 is an effective die material for low die temperatures (300-400˚C) because of its high strength and outstanding thermal shock resistance [57]. At higher temperatures (1200˚C) high pu rity dense alumina is needed [57]. Bobzin et al. [58] examined the most effective form of alumina coating, showing that using the reactive pulsed PVD process it was possible to stabilize gamma-alumina on steel substrates and obtain excellent performance for thixoforging X210CrW12 and 100Cr6. Behrens et al [59] proposed a bulk Si_3N_4 ceramic die which shows a good resistance to semi-solid steel corrosion and allows interesting surface finishing of the thixoformed parts. Unfortunately sintered ceramic tools did not show a good mechanical resistance.

Laser glazing of tool steels has been investigated [60] but the beneficial surface layer did not persist at the temperatures involved for thixoforming steels. Birol [61, 62] has investigated the performance of nickel-based IN617 and of a CrNiCo alloy as die materials but with limited success. He also showed that an increase in the die bulk temperature from 450̊C to 550˚C gives a very favourable effect from a thermal fatigue point of view [63].

Puttgen et al. [64] have investigated the thermal shock loading on coated samples, indicating a clear dependence on the base material. The combination of Ni alloy 2.4631 and HVOF gave the best results.

A self-heating ceramic tool (sintered alumina with a heating coil) has been successfully developed by Muenstermann et al. [65] for thixoextrusion of X210CrW12. Almost isothermal processing can be achieved.

6 Post-forming heat treatment and parts properties

The main steels used allow the required metallurgical structures and mechanical properties to be consistently obtained with controlled cooling from the thixoforged state or a heat treatment after the forming operation [66]. Metallographic analysis, hardness measurements and fatigue and tensile testing have been carried out on as-thixoformed parts and post heat-treated parts and using different steel grades.

The microstructure of the thixoformed parts is very sensitive to the process parameters. As these parameters are coupled together, a clear view on the impact of each parameter is very difficult to determine. Nevertheless, an improvement of the part quality is clearly observed with adaptation of the composition of the steel grade and with heat treatment or controlled cooling after thixoforming.

The pre and post treatment of the thixoformed parts in terms of heating conditions and controlled cooling has a great influence on the final microstructure and the properties of the parts and their homogenization.

Puttgen et al. [67] have summarised the experience of various workers in thixoforming finished parts including a catalogue of the defects and giving suggestions for how to avoid them. Runs with hundreds of parts in them have been produced with various alloys but reproducibility has tended to be poor. They conclude that efforts are still needed to produce defect-free components in serial production at competitive costs. Solid-liquid segregation, in particular, causes major variations in carbon content through a component and thus inhomogeneous mechanical properties. Parts with great hardness and wear resistance can be produced [68-70] but there is a tendency to brittleness [71]. There is clearly further work to be done on optimising heat treatment [72]. The effects of post treatment on defects improvement have been reported also by Rassili et al. [66].

In this field there is still a large need for research to set a complete processing map that leads to better understanding of all the steel thixoforming process parameters and their possible impact on the part microstructure and thus its mechanical properties.

7 Conclusions

Semi-solid processing of steel is a very active area for research and development. Parts have been successfully made (see Figure 5) [73].

However, the main challenges now are with die protection from degradation and with industrialisation. Considerable progress has been made [74].

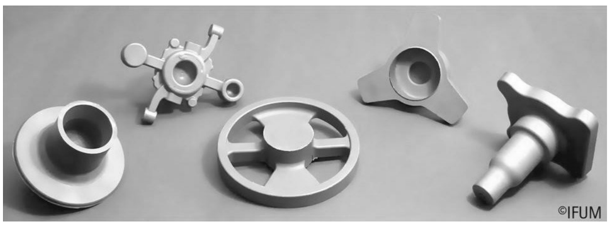

Fig. 5. Parts made in the European Project 'Adapted Steel Parts' by a collaboration of IFUM (Hannover), University of Liege and Ascometal.

References

[1] SPENCER D B, MEHRABIAN R, FLEMINGS M C. [J]. Metall. Trans., 1972, 3, 1925-32.

[2] YOUNG K P, RIEK R G, FLEMINGS M C. [J]. Metals Technology, 1979, 6, 130-7.

[3] KIRKWOOD D H. [P]. European patent No. 0305375 Thixotropic

materials, 1988.

[4] LEATHAM A, OGILVY A, CHESNEY P, WOOD J V. [J]. Metals and Mater., 1989, 5, 140-3.

[5] BOYED L E, KIRKWOOD D H, SELLARS C M. [C]. Proc. 2nd World Basque Congress on New Structural Materials, Spain, 285-95, 1988 (Publ. Vitoria Gasteiz).

[6] KAPRANOS P, KIRKWOOD D H, SELLARS C M. [J]. Proc. Instn. Mech. Engrs. Part B: J. of Engineering Manufacture, 1993, 207, 1-8.

[7] KAPRANOS P, GIBSON R C, KIRKWOOD D H, HAYES P J, SELLARS C M. [J]. Mater. Sci. & Technol., 1996, 12, 274-8.

[8] PRANGE R, NEUSCHUTZ D. [C]. Proc. 5th Int. Conf. Semi-Solid Processing of Alloys and Composites, BHASIN A K, MOORE J J, YOUNG K P, MIDSON S, (Eds.), publ. Colorado School of Mines, 653-60, 1998.

[9] MEYER S, BLECK W. [C]. op. cit. [8] 361-9.

[10] CABEZA M M, VERDEJA J I, PERO-SANZ J A, PLAZA D. [C]. Proc. 30th Annual Technical Meeting of the International Metallographic Society, Seattle, WA, Publ. ASM International, 425-8, 1997.

[11] KIM K B, LEE H I, MOON H K. [C]. op. cit. [8] 415-22.

[12] SONG R, KANG Y, ZHAO A. [J]. J. Mater. Process. Technol., 2008, 198, 291-9.

[13] WALMAG G, NAVEAU P, RASSILI A, SINNAEVE M. [C]. Proc. 10th Int. Conf. Semi-Solid Processing of Alloys and Composites, Hirt, G., Rassili, A., Buhrig-Polaczek, A. (Eds.), publ. Trans Tech Publications Ltd., Solid State Phenomena, 2008, 141-143, 415-20.

[14] SEIDL I, KOPP R. [J]. Steel Res. Int., 2004, 75, 545-51.

[15] HIRT G, SHIMAHARA H, SEIDL I, KUTHE F, ABEL D, SCHONBOHM A. [J]. CIRP Annals-Manufacturing Technology, 2005, 54, 257-60.

[16] RAMADAN M, TAKITA M, NOMURA H, EL-BAGOURY N. [P]. Mater. Sci. & Engng. A, 2006, 430, 285-91.

[17] PAHLEVANI F, NILI-AHMADABADI M. [J]. Int. J. Cast Metals Research, 2004, 17, 157-61.

[18] European Patent 0745694A1, Method and apparatus of shaping semisolid metals, UBE Industries, 1996.

[19] HAGA T, KOUDA T, MOTOYAMA H, INOUE N, SUZUKI S. [C]. Proc. ICAA7, Aluminium Alloys: Their Physical and Mechanical Properties, Vol. 1, 1998, 327-32.

[20] MILOSTEAN D C, IOAN I. [J]. Metalurgia International, 2008, 13, 49-54.

[21] RUYS A J, KERDIC J A, SORRELL C C. [J]. J. Mater. Sci., 1996, 31b, 4347-55.

[22] BULTE R, BLECK W. [J]. Steel Res. Int., 2004, 75, 588-92.

[23] OMAR M Z, ATKINSON H V, PALMIERE E J, HOWE A A, KAPRANOS P. [J]. Steel Res. Int., 2004, 75, 552-60.

[24] OMAR M Z, PALMIERE E J, HOWE A A, ATKINSON H V, KAPRANOS P. [J]. Mater. Sci. Engng. A, 2005, 395, 53-61.

[25] KAZAKOV A A. [J]. Adv. Mater. Process., 2000, 3, 31-4.

[26] FRAIPONT C, LECOMTE-BECKERS J. [C]. op. cit. [13] 523-7.

[27] LECOMTE-BECKERS J, RASSILI A, ROBELET M, PONCIN C, KOEUNE R. [C]. Proc. 9th Int Conf. Semi-Solid Processing of Alloys and Composites, Kang, C.G., Kim, S.K., Lee, S.Y. (ed.), publ. Trans Tech Publications, Solid State Phenomena, 2006, 116-117, 54-7.

[28] ROBELET M, RASSILI A, FISCHER D. [C]. op. cit. [27] 712-6.

[29] PUTTGEN W, BLECK W. [J]. Steel Res. Int., 2004, 75, 531-6.

[30] PUTTGEN W, HALLSTEDT B, BLECK W, UGGOWITZER P J. [J]. Acta Mater., 2007, 55, 1033-42.

[31] HALLSTEDT B, BALTICHEV E, SHIMAHARA H, NEUSCHUTZ D. [J]. ISIJ International, 2006, 46, 1852-7.

[32] LI J Y, SUGIYAMA S, YANAGIMOTO J, CHEN Y L, FAN G W. [J]. J. Mater. Process. Technol., 2008, 208, 165-70.

[33] ATKINSON H V. [J]. Prog. Mater. Sci., 2005, 50, 341-412.

[34] ATKINSON H V. [B] (Ed.), Modelling Semi-Solid Processing, Shaker Verlag Aachen, 2008.

[35] MODIGELL M, PAPE L, MAIER H R. [C]. op. cit.[27] 606-9.

[36] CEZARD P, FAVIER V, BIGOT R. [C]. Proc. 10th ESAFORM Conf., AIP Conf. Proc. ,2007, 907, 1149-54.

[37] OMAR M Z, ATKINSON H V, PALMIERE E J, HOWE A A, KAPRANOS P. [C]. op. cit. [27] 677-80.

[38] LI J Y, SUGIYAMA S, YANAGIMOTO J. [J]. J. Mater. Process. Technol., 2005, 161, 393-406.

[39] SHIMAHARA H, BAADJOU R, KOPP R, HIRT G. [C]. op. cit. [27] 189-92.

[40] SOLEK K P, MITURA Z, KARBOWNICZEK M, KAPRANOS P, KUZIAK R, DUTKIEWICZ J. [C]. op. cit. [13] 325-30.

[41] KANG Y L, SONG R B, LI J G, ZHAO A M. [C]. Proc. 5th Pacific Rim Int. Conf. on Advanced Materials and Processing, Materials Science Forum, Parts 1-5, 2005, 475-479, 2579-82.

[42] RASSILI A, ADAM L, FISCHER D, ROBELET M, DEMURGER J, CUCATTO A, KLEMM H, WALKIN B, KARLSSON M, FLUß A. [C]. 'Improvement of Materials and Tools for Thixoforming of Steels'. Proc. 8th Int. Conf. on Semi-Solid Processing of Alloys and Composites S2P, September 2004, Cyprus, published on CD-ROM.

[43] AHMED R., MARC, R., REGIS, B. (In fact this paper has been referenced incorrectly on Web of Science and should have as authors RASSILI, A., ROBELET, M., BIGOT, R.,) [C]. op. cit. [13] 213-8.

[44] SCHONBOHM A, GASPER R, ABEL D. [C]. op. cit.[27]734-7.

[45] BEHRENS B A, FISCHER D, RASSILI A. [C]. op. cit.[13]121-6.

[46] RASSILI A, ROBELET M, FISCHER D. [C]. op. cit. [27]717-20.

[47] BAADJOU R, KNAUF F, HIRT G. [C]. op. cit. [13] 37-42.

[48] CEZARD P, BIGOT R, BECKER E, MATHIEU S, PIERRET J C, RASSILI A. [C]. op. cit. [36] 1155-60.

[49] PIERRET J C, PhD Thesis, « la robustesse du procédés de thixoformage des aciers », Editions University of Liège, 2009.

[50] KUTHE F, SCHONBOHM A, ABEL D, KOPP R. [J]. Steel Res. Int., 2004, 75, 593-600.

[51] SIEGERT K, MESSMER G. [J]. op. cit. [50] 601-6.

[52] ABDELFATTAH S, ROBELET M, RASSILI A, BOBADILLA M. [C]. Proc. 6th Int. Conf. Semi-Solid Processing of Alloys and Composites, Chiarmetta G L, Rosso M (Eds.), Turin, Italy, September 27-29, 2000, publ. Edimet Spa, Brescia, pp. 283-8.

[53] LUGSCHEIDER E, HORNIG T, KOPP R, KALLWEIT J, MOLLER T. [J]. Adv. Engng. Mats., 2001, 3, 998-1001.

[54] KURAPOV D, SCHNEIDER J M. [J]. op. cit. [50] 577-80.

[55] BEHRENS B A, HALLER B, FISCHER D. [J]. op. cit. [50]561-8.

[56] KOPP R, SHIMAHARA H, SCHNEIDER J M, KURAPOV D, TELLE R, MUNSTERMANN S, LUGSCHNEIDER E, BOBZIN K, MAES M. [J]. op. cit. [50] 569-76.

[57] MUNSTERMANN S, TELLE R. [J]. Material Wissenschaft und Werkstofftechnik, 2006, 37, 324-8.

[58] BOBZIN K, LUGSCHEIDER E, MAES M, IMMICH P. [C]. op. cit. [27] 704-7.

[59] BEHRENS B A, FISCHER D, HALLER B, RASSILI A, PIERRET J C, KLEMM H, STUDINSKI A, WALKIN B, KARLSSON M, ROBELET M, NATALE L, ALPINI F. [J]. Solid State Phenom., 2006, 116-117, 686-689.

[60] BRABAZON D, NAHER S, BIGGS P. [C]. op. cit. [13] 255-60.

[61] BIROL Y. [J]. Ironmaking & Steelmaking, 2009, 36, 555-60.

[62] BIROL Y. [J]. Steel Res. Int., 2009, 80, 165-71.

[63] BIROL Y. [J]. Steel Res. Int., 2009, 80, 588-92.

[64] PUTTGEN W, PANT M, BLECK W, SEIDL I, RABITSCH R, TESTANI C. [J]. Steel Res. Int., 2006, 77, 342-8.

[65] MUENSTERMANN S, UIBEL K, TONNESEN T, TELLE R. [J]. J. Mater. Process. Technol., 2009, 209, 3640-9.

[66] RASSILI, A, PIERRET J C, VANEETVELD G, CEZARD P, BIGOT R, ROBELET M. [C]. Proc. 10th Int. Conf. Semi-Solid Processing of Alloys and Composites, HIRT G., RASSILI A., BUHRIG-POLACZEK A. (editors), publ. Trans Tech Publications Ltd., Solid State Phenomena, 2008, 141-143, 689-694.

[67] PUTTGEN W, BLECK W, HIRT G, SHIMAHARA H. [J]. Adv. Engng. Mats., 2007, 9, 231-245.

[68] UHLENHAUT D I, KRADOLFER J, PUTTGEN W, LOFFLER J F, UGGOWITZER P J. [J]. Acta Materialia, 2006, 54, 2727-34.

[69] DZIALLACH S, PUETTGEN W, BLECK W. [C]. op.cit.[13] 695-700.

[70] MUNSTERMANN S, TELLE R. [J]. op. cit. [62] 171-6.

[71] OMAR M Z, ATKINSON H V, KAPRANOS P, JAHARAH A G. [C]. op. cit. [36] 1185-90.

[72] RASSILI A, PIERRET J C, VANEETVELD G, CEZARD P, BIGOT R, ROBELET M. [C]. op. cit. [13] 689-94.

[73] ATKINSON H V, RASSILI A [C]. Proc. 13th ESAFORM Conference April 7-9, 2010, Brescia, Italy, publ. Springer on CD-ROM.

[74] ATKINSON H V, RASSILI A, Thixoforming Steel, publ. Shaker Verlag, Aachen, 2010.