ANNALS OF FACULTY ENGINEERING HUNEDOARA
– INTERNATIONAL JOURNAL OF ENGINEERING
Tome IX (Year 2011). Extra Fascicule. (ISSN 1584 – 2673)

1. Daniela MILOŞTEAN, 2. Mihaela FLORI

AN OVERVIEW ON THE SEMISOLID STATE PROCESSING OF STEEL

1-2. FACULTY OF ENGINEERING HUNEDOARA, UNIVERSITY "POLITEHNICA" TIMISOARA, ROMANIA

ABSTRACT: The high melting point alloys such as steels are difficult to process in semisolid state due to high temperature needed for processing. Owing to the obvious advantages of this new technology comparative with the conventional manufacturing technologies, many studies try to resolve the technological problems and to offer the possibility of a profitable commercial exploitation. This article emphasizes the importance of solid fraction in semisolid state processing technology and exposes the main steel grades investigated and presented in the literature. Also are presented the methods to obtain precursory material for thixoforming and the materials used for die manufacturing needed in this new process.
KEYWORDS: steel, semisolid state, solid fraction, thixoforming, die material

INTRODUCTION

Topics as decreasing manufacturing costs and increasing the quality of the final products draw attention to many production engineers. Thus, the development of new technologies as *semisolid state processing* (known as SSP) becomes necessary.

Semisolid processing of alloys was initiated at the beginning of the 70's at MIT (Massachusetts Institute of Technology) [1,2]. At the basis of this new technology are the experiments realized by David Spencer *et al.* on Sn-15Pb alloy were he obtained a semisolid suspension with thixotropic characteristics by mechanical stirring [3]. Semisolid metal forming is realized at temperatures between liquidus and solidus lines, when in the alloy exist about 40-60% solid fraction [4]. In this range, if the microstructure of the alloy is globular it manifests a property named thixotropy. This term was introduced by Peterfi in 1927 to define the property of slurry which become fluid when is agitated and to thicken when resting [5]. Thus, this interesting property of semisolid alloys with globular structure made possible the invention of a new technology that offers several advantages over the conventional casting, forging, etc, such as porosity reduction, lower forming temperatures, improved flow properties, reduced process force, near net shape forming, better mechanical properties, etc. [6]. The most known SSP technologies are: *thixoforming* and *rheocasting* used for aluminum and other alloys, respectively *thixomolding* and *rheomolding* especially for magnesium alloys [7]. Thixoforming consist in obtaining parts in final shape from semisolid slurries with globular structure achieved by heating the semi-finished product with non-dendritical structure. If the shaping takes place in an open die the process is called thixocasting and if the shaping is realized in a close die, is called thixoforging. Rheocasting is actually the process of forming in the semisolid state direct from cooling melt, without the need for using of a globular structure semi-finish product [3].

This article presents an overview on the most studied steels processed in semisolid state; it also refers on the methods used to obtain precursor material for thixoforming and on the most suitable materials for die manufacturing.

THE IMPORTANCE OF SOLID FRACTION IN SSP

The solid fraction is one of the most important parameters that affect principally the viscosity of the semisolid slurry. It can be calculated using a simple phase diagram (fig.1) and level rule. In fig. 1 is presented the phase diagram of a binary alloy, together with microstructures obtained by casting (fig.1.a) and by thixoforming (fig.1.b) of X210CrW12 steel. So, for simple binary eutectic alloys that melt and solidify under equilibrium condition (assuming that the liquidus and solidus line are linear), the weight of solid fraction *fs* at a given processing temperature *T* is given by [8,9]:

$$f_S^{ech} = \frac{(T_M - T) - m_L \cdot c_0}{(T_M - T)\cdot(1-k)} \quad (1)$$

where T_M is the melting point of the pure solvent, m_L is the slope of the liquidus line, c_0 is the alloy composition and k is the partition coefficient of the alloy.

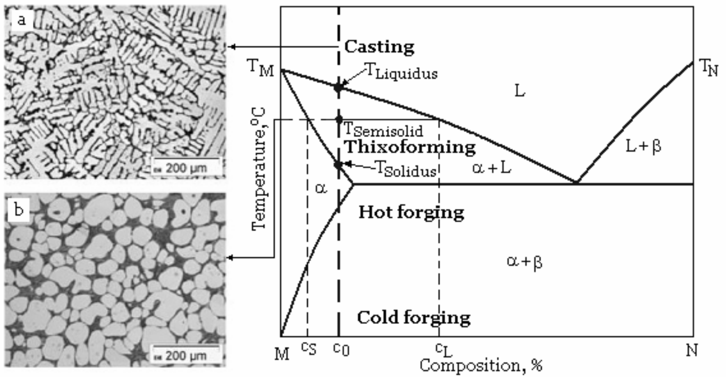


Fig.1. Schematic representation of the thixoforming area in a simple phase diagram and the corresponding microstructure a-dendritic, b-globular) for X210CrW12 steel [10]

Under fast solidification condition, when occurs a complete diffusion in the liquid and no diffusion in the solid phase (maximum microsegregation) the weight of solid fraction *fs* at a given processing temperature *T* is given by Scheil equation [8,9]:

$$f_S^{Sch} = 1 - \left(\frac{T_M - T}{T_M - T_L} \right)^{-\frac{1}{1-k}} \qquad (2)$$

where T_L is the liquidus temperature of the alloy.

The steel grades processed in semisolid state are selected according to few criteria:

- ❖ the material must have a wide solidification range (T_L-T_S). Usually the methods used to determine the semisolid range are Differential Thermal Analysis-DTA (fig.2), Differential Scanning Calorimetry-DSC and thermodynamic data [9];
- ❖ the temperature processing interval when in the alloy exist 40-60% solid fraction f_S must be sufficiently large;
- ❖ the volume of solid fraction must not be sensitive to temperature variations specially in the range with 40-60% solid fraction [4,8].

It can be seen in fig.2 that the X210CrW12 steel has a temperature processing range (40-60% f_S) of 67°C while for 100Cr6 steel the interval is only of 10°C. Hence for the second steel grade it is necessary a very strict temperature control [11].

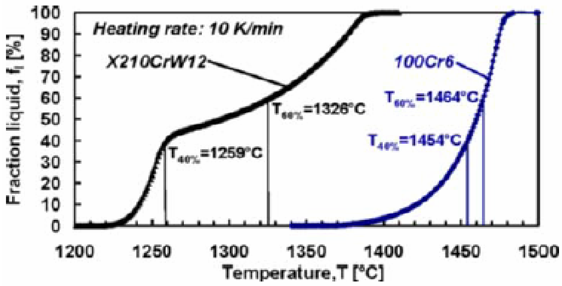


Fig.2. The variation of the liquid fraction versus temperature obtained by DTA [11]

So, the slurry temperature influences the solid fraction with direct consequences on the viscosity which indicate the capability of a material to fill a mold and determines the force necessary for deformation and flow of materials [2].

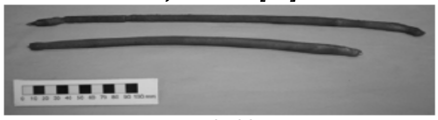
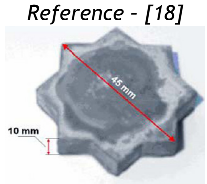
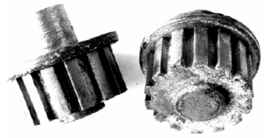
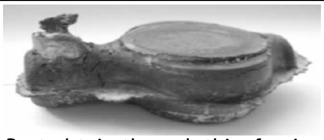
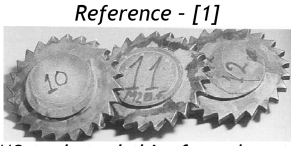
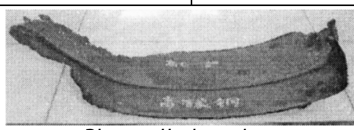
❖ STEEL GRADE SELECTED FOR THE SSP

It should be noted that semi-solid processing bases were established by studying low melting point alloys such as aluminum and magnesium alloys used for the most parts that are now commercialized. So far, the SSP technology was not applied on industrial scale for high melting point alloys such as steels [12-14], but over time have been done researches on several steel grades that are more likely to be processed by this new technology (tab.1).

At the University of Science and Technology Beijing, China, a research group have focused the studies on the rheo-rolling process and developed a device for semisolid steel preparation and rolling. The steel grades used in their investigation are principally: 60Si2Mn, 1Cr18Ni9Ti and T12 [6,21-23,26]. Results showed that semi-solid slurry obtained through electromagnetic stirring, of the steel grades 60Si2Mn and 1Cr18Ni9Ti, can be rolled successfully. They observed that for 1Cr18Ni9Ti stainless steel the strength of the plate rheo-rolled is increased compared to that of the conventionally repeated hot-rolled plate, but the elongation is decreased [22]. For 60Si2Mn spring steel rheo-rolled the rupture strength and elongation are lower than that of traditionally repeated hot-rolled plate [21]. Also, through rheo-rolling at different solid fraction, with increasing the solid fraction the mechanical properties (rupture strength and elongation) of semisolid rolled products become better [6]. T12 high carbon steel (1.2%C) was investigated only in terms of microstructure [23].

The Institute of Metal Forming, RWTH Aachen University of Technology, from Germany is another research center interested of the SSP technology. G. Hirt and his team investigated two different process routes for semi-solid precursor material preparation subsequently forged in semisolid state. The 100Cr6 steel grade was chosen for investigation because of the various application possibilities, and the X210CrW12 steel for his good suitability for microstructure studies. They concluded that the preliminary semisolid forging and rheoforging trials for both steels grade predict the high potential of the process [11]. The study realized by W. Püttgen et al. on the two steel grades mentioned above, describe the phase formation during rapid cooling from semisolid state. They have shown that the microstructure in the as-quenched state does not reflect the condition from semisolid state, and therefore is not possible a metallographic evaluation of the liquid fraction [12].

Table 1. An overview on the steel grades processed in semisolid state

X210CrW12 (AISI D6)	*Process - Thixocasting Year - 2004 Reference - [15]*  *Thixocasted steel part*	*Process -Thixo-extrusion Year - 2009 Reference - [16]*  *Extruded bars*
	Process -Thixo-extrusion Year - 2009 Reference -[17]  *Extruded bars. Left: view of a cut segment, right: cross-section*	*Process - Thixocasting Year - 2009 Reference - [18]*  *Thixocast sample*
100Cr6	*Process - Rheoforging Year - 2005 Reference - [11]*  *Part obtain through rheoforging*	*Process - Thixocasting Year - 2010 Reference - [19]*  *Thixocast samples*
	Process - Thixoforging Year - 2010 Reference - [20]  *Part obtain through thixoforging*	
M2	*Process - Thixoforging Year - 1993 Reference - [1]*  *M2 tool steel thixoforged parts*	*Process - Thixoforming Year - 2010 Reference - [14]*
60Si2Mn	*Process - Rheo-rolling Year - 2003 Reference - [21]*	*Process - Rheo-rolling Year - 2008 Reference - [6]*
1Cr18Ni9Ti	*Process - Rheo-rolling Year - 2003 Reference - [22]*	*Process - Rheo-rolling Year - 2008 Reference - [6]*
T12	*Process - Rheo-rolling Year - 2005 Reference - [23]*  *Rheo-rolled product*	
UHCS	*Process - Rheocasting Year - 2006 Reference - [24]*	
HP9/4/30	*Process - Thixoforging Year - 2005 Reference - [25]*  *Thixoforged fingers from a slug*	

In the work carried out at the University of Sheffield a research group has demonstrated the feasibility of HP9/4/30 steel thixoforging having prior microsegregated microstructure [25]. The steel grades M2 [14], X210CrW12 [18] and 100Cr6 [19] are also investigated at the Institute of Metallurgy and Materials Science of the Polish Academy of Science, Krakow.

Even if semisolid forming of steel is more difficult to be realized compared with that of low melting point alloys there is a progress made so far in this domain and exists the possibility of industrial scale implementation.

❖ METHODS USED TO OBTAIN SEMISOLID PRECURSORY MATERIAL

A successful thixoforming process requires precursory material with a unique microstructure in which the solid particles are spheroidal. This material has a thixotropic behavior in semisolid state and it can be obtained in several ways as were published in the literature [7,27].

The usual routes used to produce the steel feedstock material for thixoforming are: electromagnetic stirring (fig.3), spray forming-Osprey (fig.4) and SIMA-Strain Induced Melt Activated. For low melting point alloys besides the above mentioned methods there are other techniques of obtaining raw material for thixoforming as: mechanical stirring [28], casting at liquidus temperature (the UBE new rheocasting process is based on this principle) [27], the new MIT process [27], etc.

The *electromagnetic stirring* process was developed in the USA and consists in breaking the dendrites during solidification due to the rotating electromagnetic fields within the continuous casting crystalliser and in forming of an alloy with a nondendritic structure under the shape of a rosette [7,29]. This method is used both in the semisolid processing of low and high melting point alloys (60Si2Mn, 1CrNi9Ti [6,21,22]).

In the *SIMA process* the alloy which has been previously hot-worked is heated so recrystallistation take place before partial melting; so the liquid penetrates the grain boundaries and leads to a fine globular microstructure [27,30].

In the *OSPRAY process* the melted metal passes through a nozzle and encounters an inert gas at a high pressure. The liquid metal is sprayed by the high pressure gas as micrometric drops which cool down with a high speed while being in the air. While the bigger drops remain intact and the smaller ones are solidified during spraying, those having intermediary dimensions become semisolid (fig.4). Liquid and semisolid drops with a high liquid fraction spread upon impact, while the solid and semisolid drops with a high solid fraction get fragmented. So the resulted structure contains fine and spherical grains [27].

In 1993 Kapranos and his colleagues [1] have investigated two distinct routes (RAP and Ospray) used to produce the feedstock for M2 tool steel thixoforging. Both methods have proved to be viable for production of precursory material for tool steel thixoforging.

Recently feedstock material (M2 tool steel) obtained by Ospray method was thixoformed and the results show that the hardness value recorded a significant increase [14].

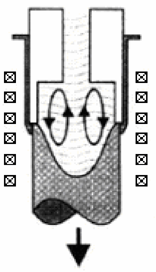


Fig.3. Electromagnetic stirring [29]

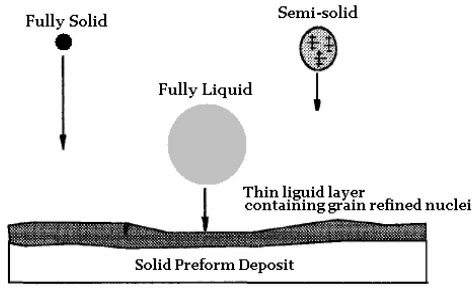


Fig.4. The Osprey process [1]

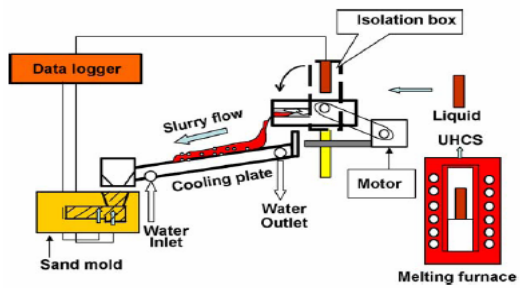


Fig.5. The cooling plate technique [24]

The "rheo" routes (rheocasting, rheoforging, rheo-rolling, rheoextrusion) does not require semi-finished products with globular structure, because in this type of process the melted material is subjected to different techniques (electromagnetic stirring [21-23], mechanical vibration [31], cooling slope technique - fig.5 - [24,32], etc.) in order to obtain in the semisolid interval a globular solid fraction in a liquid matrix. Ultra-high carbon steel (UHCS) with 1.49%C processed in semisolid state by the cooling plate technique gave good results in terms of the mechanical properties compared with the conventional casting [24].

Because the rheo-route does not require a semi-finished product with a globular structure as a starting material there are lower energy costs and thus from this point of view this route is preferable.

❖ DIE MATERIALS

One of the biggest problems which appear at the semisolid state processing of steel is finding an appropriate die material. This is due to the high temperature at which the process takes place. The searched material must have properties like: resistance at high temperatures, thermal shock resistance, good wear and corrosion resistance [33], durability in exploitation, low coefficient of thermal expansion [34], etc. In this condition the materials appropriated for thixoforming die are principally dense ceramic materials, laser treated steels or others alloys (Inconel 617, Satellite 6, CrNiCo).

Recently, S. Muenstermann et al. developed a self-heating ceramic tool for the semi-solid extrusion of steel (X210CrW12) under near-isothermal conditions. The ceramic material used for the die is high-purity alumina (Al_2O_3). Results showed that this new concept allows isothermal thixoextrusion of the steel. Also they investigated the behavior of the alumina die regarding wear and corrosion resistance. The conclusion was that by applying the self-heating tool concept the ceramic dies have excellent corrosion and wear resistance, and regarding the chemical interaction between tool and the work material, alumina dies are not decomposed in the steel thixoextrusion process, leaving the work piece unaffected [17,33].

Another potential tool material for steel semisolid forming is silicone nitride (Si_3N_4) thanks to its high strength and excellent thermal shock resistance at 300-400°C [35]. The non-oxide ceramics materials (Si_3N_4) are susceptible to oxidation and corrosion while oxide ceramics (Al_2O_3) exhibit significant lower mechanical properties and poor thermal shock resistance [35]. Despite these properties unfavorable for a die material used in semisolid processing of steel, S. Muenstermann and R. Telle developed two new tool concept strategies based on the benefits of the both materials (high strength of non-oxide ceramics materials and excellent corrosion resistance of oxide ceramics) [35]. A schematic view of the tool concept strategies for thixoforming dies is given in fig.6.

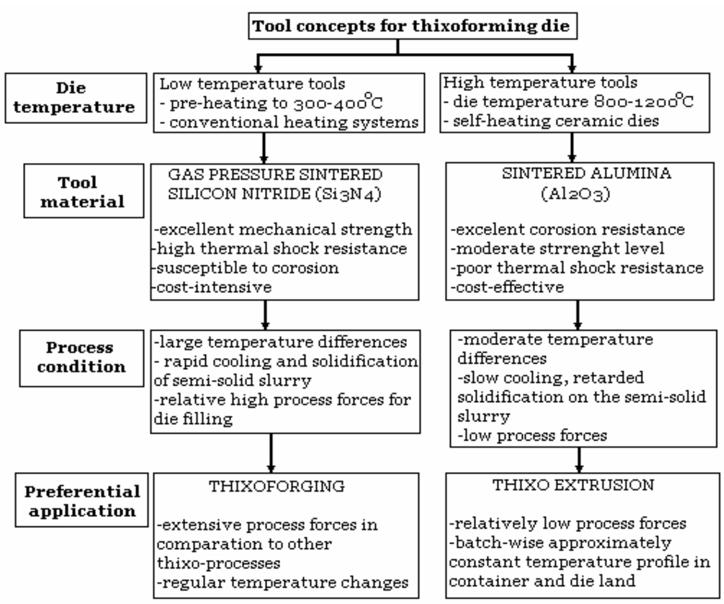


Fig.6. Tool concept strategies for steel thixoforming die [35]

These two strategies developed by S. Muenstermann and R. Telle for the thixoforging and thixoextrusion of steel using silicon nitride and respectively alumina die were experimented and the results were promising.

Regarding the die final price, it was concluded that the alumina tool costs (row material, production and investment costs) are lower compared with the non-oxide ceramics [33].

Y. Birol [34] tested the CrNiCo die material alloy and used it in the steel thixoforming process. By comparing the thermophysical properties and the thermal fatigue behavior of CrNiCo alloy with the X38CrMoV5 steel used in manufacture of conventional forging dies, it was concluded that the first one can be a viable die material.

Other alloys (Inconel 617, Satellite 6) were investigated as die materials and subjected to high temperature abrasive wear testing and compared to the X32CrMoV33 hot work tool steel (used in conventional hot forging of steel parts). At 625°C, the wear resistance (represented by weight loss) and the surface hardness of the tool steel is better compared to the alloys, while at 750°C the results are superior for the alloys [36]. From the point of view of service life, Inconel 617 and Satellite 6 alloys presented few and shallow cracks after 5000 thermal cycling wile X32CrMoV33 steel resisted only 1500 cycles [37]. Also, by laser glazing of tool steel (AISI H13) dies, the initial results showed a large increase in hardness value for the surface layer, but the temperature working limit was only of ~600°C. So the results are unsatisfactory for thixoforming of steel (where the die cavity surface reaches 700-750°C only for a few seconds [34,38]) and this steel could by used as die material for non-ferrous semisolid forming [39,40].

The investigations realized so far on die materials are encouraging, but just now it wasn't found a material that meets all requirements necessary for large scale use.

❖ CONCLUSIONS

Analyzing the literature studies it may be concluded that the semisolid processing has many advantages over traditional technologies and has already been applied to industrial production of magnesium and aluminum alloys. In recent years, steel semisolid processing also drew attention, the results on applying this technology being encouraging with high melting point alloys.

Also finding an appropriate die material with a good durability in exploitation is yet under investigation, but the research made so far are promising.

❖ REFERENCES

[1.] P. Kapranos, D.H. Kirkwood and C.M. Sellars, Semi-solid processing of tool steel, Journal de Physique IV, Colloque C7, supplement au Journal de Physique III, Vol. 3 (1993)
[2.] O. Lashkari, R. Ghomashchi, The implication of rheology in semi-solid metal processes: An overview, Journal of Materials Processing Technology, Vol. 182 (2007) 229–240

[3.] M.C. Flemings, Semi-solid forming: the process and the path forward, Metallurgical Science and Technology, Vol. 18 (2) (2000)
[4.] D.I. Ulenhaut et al., Structure and properties of a hypoeutectic chromium steel processed in the semi-solid state, Acta Materialia, Vol. 54 (2006) 2727-2734
[5.] F. Czerwinski, The Basics of Modern Semi-Solid Metal Processing, JOM, Vol. 58 (6) (2006) 17-20
[6.] R. Song, Y. Kang and A. Zhao, Semi-solid rolling process of steel strip, Journal of Materials Processing Technology Vol. 198 (2008) 291-299
[7.] Z. Fan, Semisolid metal processing, International Materials Reviews, Vol. 47 (2) (2002)
[8.] E. Tzimas, A. Zavaliangos, Materials Selection for Semisolid Processing, Materials and Manufacturing Processes, Vol.14 (1999)
[9.] E. Tzimas, A. Zavaliangos, Evaluation of volume fraction of solid in alloys formed by semisolid processing, Journal of Materials Science, Vol. 35 (2000) 5319-5329
[10.] H. Stankova et al., Influence of thixoforming on structure development of the tool steel, 12th International Research/Expert Conference "Trends in the Development of Machinery and Associated Technology" TMT 2008, Istanbul, Turkey
[11.] G. Hirt et al., Semi-Solid Forging of 100Cr6 and X210CrW12 Steel, CIRP Annals – Manufacturing Technology, Vol. 54, Issue 1 (2005) 257-260
[12.] W. Püttgen et al., On the microstructure formation in chromium steels rapidly cooled from the semisolid state, Acta Materialia, Vol. 55 (2007) 1033-1042
[13.] R. Kopp et al., Forming and joining of commercial steel grades in the semi-solid state, Journal of Materials Processing Technology 130-131 (2002) 562–568
[14.] J. Dutkiewicz et al., Thixoforming of spray formed M2 tool steel, International Journal of Material Forming Vol. 3, Suppl.1 (2010) 755 – 758, DOI 10.1007/s12289-010-0880-3
[15.] H. Bramann et al., Casting of a cold work steel alloy in semi-solid state, Journal of Materials Processing Technology 155-156 (2004) 1357-1364
[16.] F. Knauf, R. Baadjou and G. Hirt, Analysis of semi-solid extrusion products made of steel alloy X210CrW12, International Journal of Material Forming, Vol. 2, Suppl. 1 (2009) 733-736 DOI 10.1007/s12289-009-0426-8
[17.] S. Muenstermann et al., Semi-solid extrusion of steel grade X210CrW12 under isothermal conditions using ceramic dies, Journal of Materials Processing Technology 209 (2009) 3640–3649
[18.] J. Dutkiewicz et al., Thixoforming technology of high carbon X210CrW12 steel, International Journal of Material Forming Vol. 2, Suppl. 1: 753 – 756 (2009), DOI 10.1007/s12289-009-0558-x
[19.] L. Rogal et al., Characteristics of 100Cr6 bearing steel after thixoforming process performed with prototype device, Trans. Nonferrous Met. Soc. China 20 (2010) 1033-1036
[20.] K. P. Solek et al., Characterization of thixoforming process of 100Cr6 steel, Trans. Nonferrous Met. Soc. China 20 (2010) 916-920
[21.] W. Mao et al., Slurry Preparation and Rolling of Semi-Solid 60Si2Mn Spring Steel, Journal of Material Science and Technology, Vol. 19, No. 6 (2003)
[22.] W. Mao et al., Semi-solid slurry preparation and rolling of 1Cr18Ni9Ti stainless steel, Journal of University of Science and Technology Beijing, Vol. 10, No.6 (2003)
[23.] J.G. Li et al., Microstructural morphology of the semi-solid high carbon steel T12 before and after rheo-rolling, Journal of University of Science and Technology Beijing, Vol. 12, No. 2 (2005)
[24.] M. Ramadan et al., Semi-solid processing of ultrahigh-carbon steel castings, Material Science and Engineering A 430 (2006) 285-291
[25.] M.Z. Omar et al., Thixoforming of a high performance HP9/4/30 steel, Materials Science and Engineering A 395 (2005) 53-61
[26.] R. Song et al., Investigation of the Microstructure of Rolled Semi-Solid Steel, Journal of Materials Sciences and Technology, Vol. 18, No. 3, (2002)
[27.] H.V. Atkinson, Modelling the semisolid processing of metallic alloys, Progress in Materials Science, No. 50 (2005) 341-412
[28.] M. Modigell, J. Koke, Time-Dependent Rheological Properties of Semisolid Metal Alloys, Mechanics of Time-Dependent Materials 3 (1999) 15-30
[29.] D. Stoica, I. Ilca, Reflection regarding the obtaining of precursor material at semisolid state procesing, Scientific Bulletin of the „Politehnica" University of Timisoara, Transaction of Mechanics, Editura Politehnica, Timişoara, ISSN 1224-6077, Tom 52(66), Fasc. 8, (2007) 43-48
[30.] J.-Y. Li et al., Microstructural evolution and flow stress of semi-solid type 304 stainless steel, Journal of Materials Processing Technology, 161 (2005) 396-406
[31.] D. Miloştean, M. Flori, Experimental research in pilot phase regarding the semisolid state processing of steel, Annals of the Faculty of Engineering Hunedoara, Mirton Publishing House, Timisoara, ISSN 1584-2673, Tome VII, Fascicule 3, (2009) 383-385
[32.] B. Amin-Ahmadi, H. Aashuri, Semisolid structure for M2 high speed steel prepared by cooling slope, Journal of Materials Processing Technology, 210 (2010) 1632-1635
[33.] S. Muenstermann, R. Telle, Wear and corrosion resistance of alumina dies for isothermal semi-solid processing of steel, Wear 267 (2009) 1566–1573
[34.] Y. Birol, Testing of a novel CrNiCo alloy for tooling application in semi-solid processing of steel, International Journal of Material Forming (2010) 3:65–70, DOI 10.1007/s12289-009-0418-8
[35.] S. Muenstermann, R. Telle, Ceramic tool concepts for the semi-solid processing of steel alloys, Mat.-wiss. u. Werkstofftech., Vol. 37, No. 4 (2006)
[36.] Y. Birol, High-temperature abrasive wear testing of potential tool materials for thixoforming of steel, Tribology International 43 (2010) 2222-2230
[37.] Y. Birol, Ni- and Co-based superalloys as potential tool materials for thixoforming of steel, International Journal of Material Forming, Vol. 3, Suppl. 1 (2010) 739-742
[38.] A. Rassili et al., X38CrMoV5 hot-work tool steel as tool material for thixoforming of steel: Numerical and experimental evaluation, Trans. Nonferrous Met. Soc. China 20 (2010) 713-718
[39.] D. Brabazon et al., Glazing of tool dies for semi-solid steel forming, International Journal of Material Forming, Vol. 2, Suppl. 1 (2008) 985-988, DOI 10.1007/s12289-008-0223-9
[40.] S.N. Aqida et al., Thermal stability of laser treated die material for semi-solid material forming, International Journal of Material Forming, Vol. 2, Suppl. 1 (2009) 761-764