INFLUENCE OF HEAT TREATMENT ON THE MICROSTRUCTURE AND TOUGHNESS OF BÖHLER M333 ISOPLAST STEEL

J. Perko, C. Redl, H. Leitner

In this work the through hardenability and the influence of the heat treatment parameters (austenitizing temperature, cooling parameter λ and tempering temperature) on the microstructure and the achievable toughness level of Böhler M333 ISOPLAST are investigated. The results are compared to the standardized tool steel grade DIN 1.2083. The investigations showed that the cooling parameter λ has a strong influence on the impact toughness of M333 ISOPLAST plastic mould steel. The toughness is reduced by pro-eutectoid precipitates and not by a lack of through hardenability. Furthermore, it was found out that depending on the cross section of the moulds appropriate heat treatments lead to a good combination of hardness and toughness.

KEYWORDS: plastic mould steel, heat treatment, toughness, through hardenability, pro-eutectoid precipitates

INTRODUCTION

Plastic mould steels which are currently available on the market, e.g. DIN 1.2083, 1.4028 and 1.2316, are often not able to fulfil the high requirements of the plastics processing industry. For that reason, Böhler Edelstahl developed the pressure-electro-slag remelted, nitrogen alloyed tool steel grade M333 ISOPLAST. Nitrogen has a lot of positive effects on martensitic chromium steels [1, 2]. The partial replacement of carbon by nitrogen leads to an increase in corrosion resistance and toughness. General corrosion is reduced as well as pitting and crevice corrosion. The improvement in toughness results primarily from the very homogeneous distribution of fine precipitates [3]. Thus, M333 ISOPLAST combines excellent mirror finish polishability with highest cleanliness and toughness levels and excellent corrosion properties.
However, as a consequence of the growing demand for large moulds, plastic mould steels must also exhibit an excellent through hardenability in order to avoid the formation of bainite or pearlite during quenching. Additionally, due to lower cooling rates a low tendency to form grain boundary precipitates, which cause grain boundary embrittlement, is required.
Therefore, this work concentrates on the investigation of the through hardenability and on the influence of the heat treatment parameters on the mechanical properties and microstructure of M333 ISOPLAST.

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MATERIALS AND INVESTIGATION METHODS

Tab. 1 shows the chemical composition of M333 in comparison to M310, which approximates the standardized tool steel grade DIN 1.2083. This steel was used as a reference steel grade for the dilatometer investigations. The samples for the dilatometer investigations of M333 and M310 were manufactured from a hot-rolled and soft-annealed bar with a diameter of 86 mm. For the dilatometer experiments a quenching dilatometer Bähr Dil 805 A/D was used. To follow the evolution of the hardness of the dilatometer samples, Vickers hardness values (HV10) were measured using microhardness tester supplied by Zwick.
For the samples for impact toughness testing of M333 two slices with a thickness of 60 mm were cut from the top of a forged and soft annealed bar with the dimension 603 x 303 mm2. Then, the samples were cut in longitudinal direction at half radius and were heat treated with an oversize of 0.5 mm on every side. For the hardening of the samples a vacuum heat treatment furnace was used. Quenching was performed by using nitrogen as quenching gas.
The cooling parameter λ is defined as the cooling time from 800 to 500 °C in seconds divided by 100. The selected cooling parameters λ were adjusted with dummy samples exhibiting the same size as the test specimens. The temperature was controlled by mounting a thermocouple in a drilled hole in the centre of the dummy sample. The heat-treated samples were grinded to the final dimension of 7 x 10 x 55 mm^3. The impact tests were performed with a 450 J pendulum Roell Amsler 101. Four samples of each heat treatment were tested and the average and standard deviation were calculated. Rockwell C hardness was measured on each specimen using a hardness tester Emco-Test M4R 025 G3. The fracture surface of all tested im-

Steel grade	DIN	C	Si	Mn	Cr	V	N
M310 ISOPLAST	~1.2083	0.38	0.70	0.30	14.30	0.20	-
M333 ISOPLAST	-	0.28	0.30	0.30	13.50	-	+

Tab. 1 **Chemical composition of the tool steels investigated.**
Composizione chimica degli acciai da utensile investigati.

Austenitizing temperature [°C]	980/1020
Holding time [min]	30
Cooling parameter λ [s x 10-2]	0.5/5/8
Tempering temperature [°C]	250/300/510/520/ 530/540
Tempering time [min]	2 x 120

Tab. 2 **Applied heat treatments for impact toughness testing.**
Trattamenti termici applicati per la verifica della resilienza.

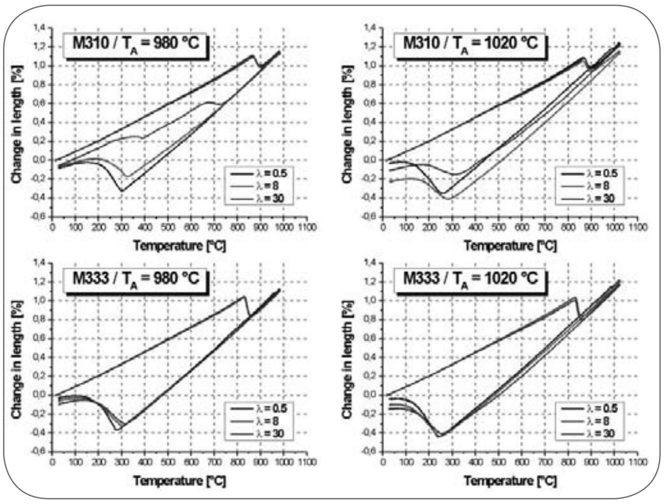

Fig. 1 **Dilatometer curves of M310 and M333.**
Curve dilatometriche degli acciai M310 e M333.

pact specimens was macroscopically documented. Additionally, the fracture surface of selected heat treatment cycles was analyzed using a scanning electron microscope Jeol JSM6460lv (SEM). Tab. 2 shows an overview of the applied heat treatments of the samples for impact toughness testing.

RESULTS AND DISCUSSION

The aim of the dilatometer investigations was to find out how the austenitizing temperature and the cooling parameter λ influence the transformation behaviour of M333 in comparison to M310. During heating and cooling of a sample in the dilatometer, the change in length (ΔL) as a function of time and temperature is recorded, from which ΔL/L0 versus temperature (T) is plotted. L0 represents the original length of the sample. Representative plots of ΔL/L0 versus T of M310 and M333 at the two austenitizing temperatures selected (980 °C and 1020 °C) are shown in Fig. 1.
It can be seen that in case of M333 for both austenitizing temperatures and all applied cooling rates only one transformation takes place during cooling. This is indicated by the distinct length change starting at about 250 °C. Microstructural investigations revealed that this length change is caused by the transformation austenite to martensite. However, the martensite start temperature (MS) is shifted to lower temperatures with increasing austenitizing temperature, which is due to the higher solution state of the austenite at higher temperatures. When M333 is austenitized at 980 °C, the remaining change in length after quenching is lower compared to 1020 °C. This indicates that the amount of retained austenite is less compared to austenitizing temperature 1020 °C, what was confirmed by XRD measurements.
In contrast, the steel grade M310 shows different transformation behaviour. At an austenitizing temperature of 980 °C and the highest investigated cooling parameter λ=30 additional transformations can be observed, starting at about 750 °C and 400 °C. Microstructural examinations of this sample showed the presence of pearlite and bainite. Thus, an austenitizing temperature of 980 °C is not suitable for parts with large cross sections made of this steel grade. For an austenitizing temperature of 1020 °C, M310 shows neither a pearlitic nor a bainitic transformation at a cooling parameter λ=30. However, for both steel grades the measured MS-temperatures are summarized in Tab. 3.
The experimentally determined hardness measurements of the dilatometer samples are also listed in Tab. 3. In case of M333 Tab. 3 reveals that while the hardness decreases with increasing λ values, the MS-temperature increases. This indicates a decreasing solid solution state with increasing λ values which might be due to the formation of pro-eutectoid phases such as carbides or carbonitrides.
The microstructure of selected dilatometer samples is shown in Fig. 2. In order to reveal the microstructure, Nital was used as etchand. In Fig. 2, the former austenite grain boundaries can

Cooling parameter λ		M333 ISOPLAST 0.5	M333 ISOPLAST 8	M333 ISOPLAST 30	M310 ISOPLAST 0.5	M310 ISOPLAST 8	M310 ISOPLAST 30
980 C	HV10	593	567	537	563	509	295
	MS [°C]	300	342	370	317	340	406*
1020 C	HV10	636	628	601	617	582	503
	MS [°C]	278	285	279	281	335	369

* BS ... Bainite Start Temperature

Tab. 3 **MS-temperature and hardness of M333 and M310 after various heat treatment cycles.**
Temperatura MS e durezza degli acciai M333 e M310 dopo differenti cicli di trattamento termico.

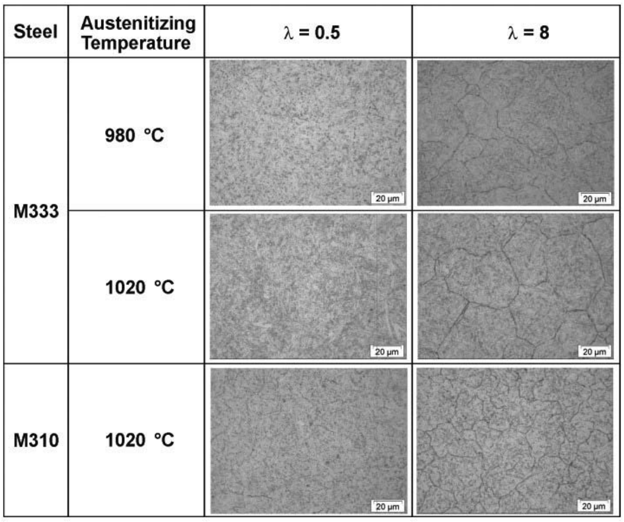

Fig. 2

Microstructure of selected heat treatment cycles.
Microstruttuta relativa ai diversi cicli di trattamento termico scelti.

not be seen in both M333 samples quenched with λ=0.5. On the other hand, the grain boundaries of M310 at an austenitizing temperature of 1020 °C are weakly visible, indicating that the grain boundaries are decorated. Additionally, M310 contains some coarser M23C6 carbides, which is one of the reasons for the significantly lower toughness level in comparison to M333, as reported in [4, 5].
At a cooling parameter λ=8 both steels show a strong decoration of the grain boundaries, which is designated by the distinct appearance of the grain boundaries. However, the strongest decoration shows M333 when it is austenitized at 1020 °C. The M333 samples austenitized at 980 °C exhibit a finer grain and the decoration of the grain boundaries is less pronounced. The reason for the lower austenite grain size of the M333 samples austenitized at 980 °C is the higher content of undissolved precipitates, which stabilize the grain size during austenitizing. At 1020 °C their amount is strongly reduced, which causes grain growth.
The reason for the decoration of the former austenite grain boundaries seems to be due to the formation of pro-eutectoid, chromium-rich precipitates as reported in [6, 7]. Herzog [7] investigated oil and air quenched samples with SANS and found out that the amount of pro-eutectoid precipitates in the size range from 1 to 100 nm increases with increasing austenitizing temperature and cooling parameter, which correlates well with the results of M333.
The results of the dilatometer investigations indicated, that the interaction of pro-eutectoid precipitations and austenite grain size may have more impact on the toughness of M333 than a lack of through hardenability, as the grain boundary decoration occurred at λ-values below 8. Therefore, the impact toughness investigation of M333 concentrated on λ-values below 8.
However, the investigations are primarily focused on the heat treatment of larger cross sections. For that reason the tempering of most samples was carried out at temperatures between 510 and 540 °C, which are higher than the secondary hardening peak of M333. Temperatures above the secondary hardening peak are needed to reduce residual stresses and retained austenite as far as

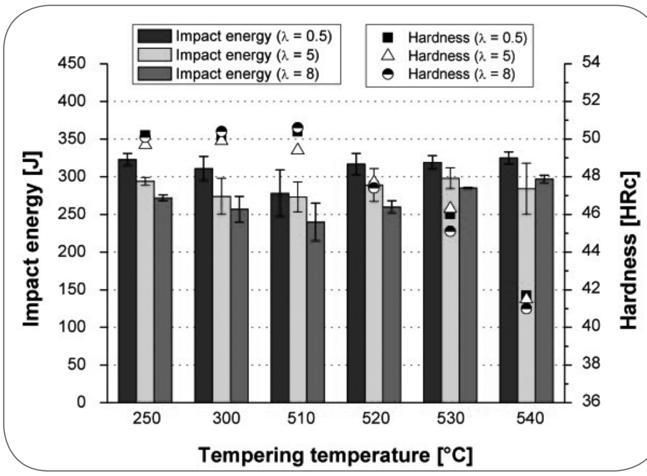

Fig. 3

Impact energy and hardness of M333, austenitized at 980 °C.
Valori di resilienza e durezza dell'acciaio M333, austenizzato a 980 °C.

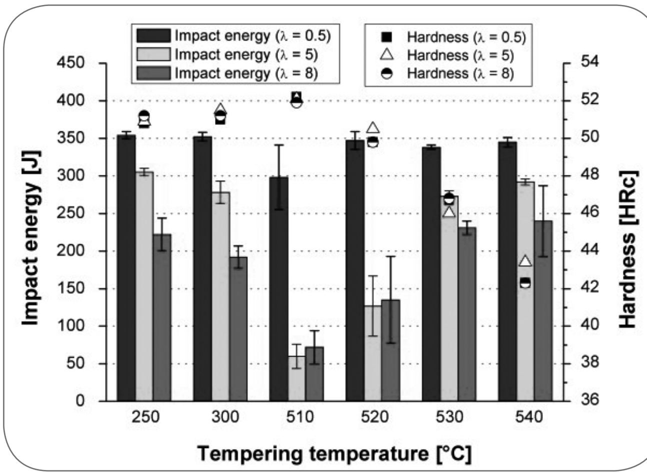

Fig. 4

Impact energy and hardness of M333, austenitized at 1020 °C.
Valori di resilienza e durezza dell'acciaio M333, austenizzato a 1020 °C.

possible. However, for comparison the tempering temperatures 250 and 300 °C were included in the investigations.
Fig. 3 and 4 show the impact energy versus tempering temperature of the samples austenitized at 980 °C and 1020 °C, respectively. Additionally, the measured hardness values are plotted in the diagrams. The results reveal that the hardness at each tempering temperature is virtually independent of the cooling parameters up to λ=8. The hardness level of the samples hardened at 980 °C is continuous 1-2 HRc lower than the hardness of samples austenitized at 1020° C. Nevertheless, a hardness of approximately 50 HRc, which is typical for the plastic moulding industry, is still achievable with an austenitizing temperature of 980 °C.
In contrast to the hardness, the achievable toughness level is strongly depending on the cooling parameter λ, especially for the samples austenitized at 1020 °C. As long as the cooling parameter λ is low (λ=0.5), the samples austenitized at 1020 °C show high hardness and toughness values in all heat treatment cycles

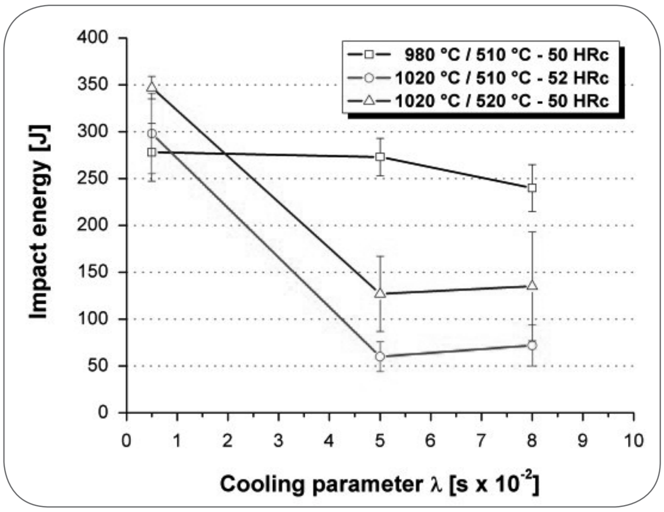

Fig. 5

Impact energy of M333 as a function of the cooling parameter λ.

Valori di resilienza dell'acciaio M333 in funzione del parametro di raffreddamento λ.

investigated. When the cooling parameter is increased to λ=5, the toughness is significantly reduced, especially for the most relevant tempering temperatures 510 and 520 °C, which have to be applied to reach hardness levels of 50 HRc at temperatures above the secondary hardening peak.
For the most relevant tempering temperatures for industrial application, the impact energy as a function of the cooling parameter λ is illustrated in Fig. 5. It can be seen that the cooling parameter λ has only little influence on the impact toughness of samples austenitized at 980 °C, which is beneficial for the heat treatment of tools with large cross sections. In contrast to that, the negative influence of the cooling parameter λ on the impact toughness of samples austenitized at 1020 °C is more pronounced. For that reason, an austenitizing temperature of 1020 °C seems to be appropriate for tools with small cross sections only.
To investigate the microstructural mechanisms that cause embrittlement of M333, the fracture surface of the tested specimens was investigated macroscopically and by means of SEM (Fig. 6 and 7).
Fig. 6 shows that the cooling parameter λ has only little influence on the fracture appearance of samples austenitized at 980°C. There is only a marginal reduction of ductility when samples with low and high cooling parameters are compared, which is in accordance with the measured impact toughness values. In contrast to that, the samples austenitized at 1020°C show a high loss of ductility with increasing λ values. This effect is extremely pronounced at tempering temperatures of 510 and 520 °C, where the fracture mode changes from ductile to mainly brittle already at λ=5. This is also reflected in the low impact toughness values measured.
A detailed fracture analysis was conducted by SEM investigations. Fig. 7 shows the fracture surface of the samples austenitized at 980 and 1020°C and tempered at 510 °C in the region of the incipient crack.
It is obvious that both samples quenched with λ=0.5 exhibit a ductile fracture, which corresponds well with the high impact toughness values measured. When the cooling parameter λ is increased up to 5 the fracture appearance changes significantly. The samples which were austenitized at 980°C still exhibit a ductile fracture, but the intergranular character increases. In contrast to that, the samples austenitized at 1020 °C and cooled with λ=5 exhibit an entire intergranular fracture with no ductile character at all, being the reason for the low impact toughness of these samples. When the cooling parameter is increased to λ=8, the intergranular character of samples austenitized at 980 °C increments, but there is still a high portion of ductile fracture visible.
A more detailed view on the surface of the fracture of the samples hardened at 1020°C with cooling parameter λ=5 confirms this theory (see Fig.8). However, the presence of pro-eutectoid precipitates could not be clearly confirmed with SEM-EDX, as the resolution of this method is too low. But it can be assumed that the formation of precipitates preferentially takes place at the grain boundaries, as the critical nucleation energy is lower there and because of the lower surface energy that needs to be brought up. Therefore, precipitations with a low coherency develop preferentially there [6].
However, the investigations revealed that a lack of through hardenability can be excluded; the embrittlement with increasing λ values can only be caused by a disadvantageous combination of austenite grain size and high solution state of the austenite. This encourages the formation of pro-eutectoid precipitates at the austenite grain boundaries at higher λ values, which causes the

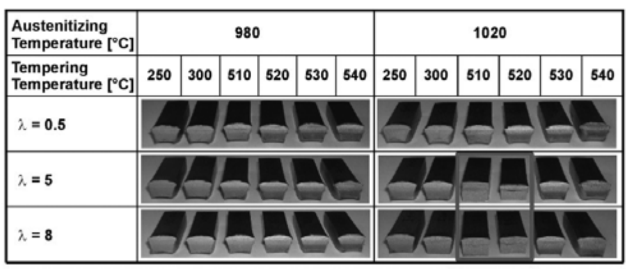

Fig. 6

Macroscopic comparison of the broken M333 samples.

Confronto macroscopico della superficie di rottura del provino in acciaio M333.

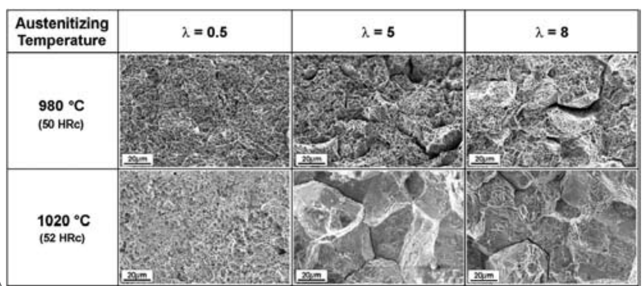

Fig. 7

SEM images of the fracture surfaces (all samples were tempered at 510 °C).

Immagini al SEM delle superfici di rottura (tutti i campioni sono stati rinvenuti a 510°C).

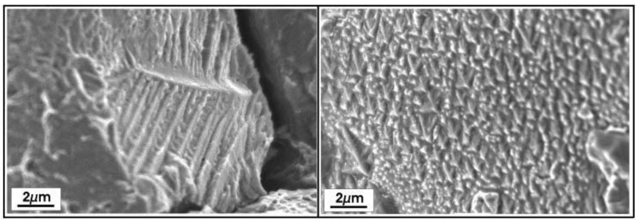

Fig. 8
Detailed view of the SEM image in Fig. 7 (1020 °C / λ=5).
Particolare dell'immagine al SEM di Fig. 7 (1020 °C / λ=5).

observed embrittlement of M333, especially in the region of the secondary hardening peak.
Additionally, heat treatment simulations were carried out in order to determine the temperature in the centre of a block during quenching. Nitrogen gas with a pressure of 5 bar was used as quenching medium. Fig. 9 depicts the temperature evolution in the centre of work pieces with different square cross sections. The results show that a cooling parameter λ of approximately 8 is achievable in the centre of a block with a dimension between 150 x 150 mm2 and 200 x 200 mm2. However, Fig. 9 also reveals that cooling of real work pieces does not follow one λ-value. At the beginning cooling is always slower, which enables the formation of pro-eutectoid precipitates on grain boundaries. This cooling characteristic also applies for the quenching of the impact toughness samples investigated. Therefore, the measured impact toughness values reflect the behaviour of real work pieces.

CONCLUSIONS

The cooling parameter λ has a strong influence on the impact toughness of M333 ISOPLAST plastic mould steel. It was found that the toughness is reduced by pro-eutectoid precipitates and not by a lack of through hardenability.
If low cooling parameters λ can be attained, e.g. during heat treatment of tools with small cross sections, austenitizing at 1020 °C results in the best combination of high hardness and impact toughness. On the other hand, the application of lower austenitizing temperatures, e.g. 980 °C, significantly reduces the negative influence of higher cooling parameters λ on the impact toughness.
This study showed that Böhler M333 ISOPLAST is a suitable steel grade for moulds with small and large cross sections, if an appropriate heat treatment is applied. However, additional work is required to gain detailed information on formation of the precipitates which cause embrittlement.

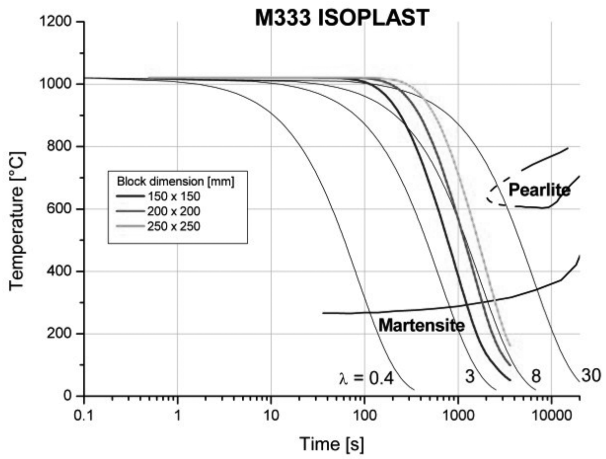

Fig. 9
Temperature evolution in the block centre for different dimensions (N2 5 bar).
Evoluzione della temperatura al centro del blocco per diverse dimensioni (N2 5 bar).

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REFERENCES

1] R. SCHNEIDER, F. KOCH, P. WÜRZINGER, Proc. of the 5th International Tooling Conference, Leoben, September 1999, p.265.
2] G. LICHTENEGGER, Proceedings of the 15th Leobener Kunststoffkolloquium, Leoben, November 1999, p.XIV-9.
3] G. LICHTENEGGER, R. SCHNEIDER, J. SAMMER, G. SCHIRNINGER, P. WÜRZINGER, J. NEUHERZ, Proc. of the 5th International Tooling Conference, Leoben, September 1999, p.644.
4] K. SAMMT, J. SAMMER, J. GECKLE, W. LIEBFAHRT, Proc. of the 5th International Tooling Conference, Karlstad University, September 2002, p.285.
5] D. UGUES, M. ROSSO, A. RIVOLTA, Proc. of the 6th International Tooling Conference, Politecnico di Torino, May 2006, p.325.
6] H. BERNS, M. POHL, Praktische Metallographie 18 (1981), p.209.
7] C. HERZOG, Master thesis, University of Leoben (1995).

ABSTRACT

INFLUENZA DEL TRATTAMENTO TERMICO SU MICROSTRUTTURA E TENACITÀ DELL' ACCIAIO BOHLER M333 ISOPLAST

Parole chiave: trattamenti termici, proprietà, acciaio

In questo lavoro sono stati analizzati temprabilità e l'influenza dei parametri di trattamento termico (temperatura di austenizzazione, parametro di raffreddamento λ, temperatura di rinvenimento) sulla microstruttura e sul livello di durezza ottenibile per l'acciaio Böhler M333 ISOPLAST. I risultati sono stati confrontati con il classico acciaio per utenisili DIN 1.2083. Le indagini hanno evidenziato che il parametro di raffreddamento λ ha una forte influenza sulla resistenza all'urto dell' acciaio M333 ISOPLAST per stampi destinati all'industria delle materie plastiche. La tenacità risulta ridotta dai precipitati pro-eutettoidici e non dalla mancanza di temprabilità. Inoltre, si è accertato che a seconda della sezione trasversale degli stampi, i trattamenti termici appropriati portano ad un buon compromesso fra durezza e tenacità.