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Production of ultra-pure steel intended for forged elements

A. Michaliszyn*, Z. Wcisło, M. Rydarowicz
Faculty of Metals Engineering and Industrial Computer Science, AGH University of Science and Technology, Al. Mickiewicza 30, 30-059 Kraków, Poland
* Corresponding e-mail address: michalis@agh.edu.pl

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ABSTRACT

Purpose: The purpose of this research is to analyse the technology of making ultra-pure steel intended for casting forging ingots. Forging ingots, whose mass amounts to 8 Mg, are cast using the bottom pouring method after vacuum steel degassing in the ladle.
Design/methodology/approach: Data from 24 melts were analysed. Researchers studied not only the final content of oxygen, sulphur and hydrogen after vacuum steel refinement but also the quantitative amount of non-metallic inclusions in forgings made of ingots. A simulation was also conducted. Its purpose was to assess the optimal share of bauxite in the production of refining slag. The simulation was performed using thermodynamic software called FactSage 5.5.
Findings: Analysing the final concentration of oxygen dissolved in liquid steel led to a conclusion that approx. 90% of analysed types of steel can be categorized as ultra-pure. The simulation results concerning refining slag formation show that the use of bauxite as a slag forming additive lead to an increase in the liquid phase, and what follows, a decrease in the share of solid precipitations – including the precipitations of lime.
Research limitations/implications: It was concluded that all stages of ultra-pure steel production must be conducted conscientiously and meticulously. It is also necessary to improve the conditions of vacuum steel refining process by equipping steelworks with a new vacuum device, e.g. of VOD type.
Practical implications: If all stages of steel making are conducted conscientiously and meticulously and the steelworks are equipped with a device for vacuum steel refinement, then the produced steel (from the point of view of quality) can be competitive in the market of the ultra-pure steel intended for forged products.
Originality/value: The simulation results concerning the production of refining slag show that the use of bauxite as a slag forming material leads to an increase in the share of liquid phase. Refining steel under a heating layer contributes to the improvement of steel purity – which is measured by analysing the share of surface non-metallic inclusions. Refining steel under refining slag with a basic character and physical properties adapted to the temperature close to the liquidus temperature of steel contributes to a decrease in the amount of non-metallic inclusions, a decrease in their size. It can aslo affect the shape of inclusions - making them almost spherical in shape. This method should be optimised and further research should be conducted into improving the purity of steel intended for forged shafts for the power industry.
Keywords: Ultra-pure steel; Oxygen; Sulphur; Hydrogen; Non-metallic inclusions; Refining slag

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1. Introduction

Due to a constant drive to decrease CO_2 emissions by increasing the amount of electric energy from renewable and unconventional sources the power industry reports high demand for bigger and bigger forged machine components (e.g. a turbine shaft, generator rotors) made of high purity steel, so called ultra-pure steel.

Obtaining high quality products in modern metallurgy consists in isolating steel refining processes from steel making furnace and moving refining processes out of the furnace into the ladle. In such a way a number of metallurgical aggregates were made: ladle furnace (LF), VOD, VAD. Steel refining process in these aggregates is very efficient and allows to obtain high purity steel.

As far as ladle metallurgy processes are concerned, properly formed refining slag is the condition of obtaining high purity steel which will meet increasingly high requirements of the recipients of steel products. This slag needs to have good desulphurization properties and it needs to assimilate non-metallic oxide inclusions.

The term “steel purity” is not an unequivocal one. It was first used during first melting processes. It referred to steel which is pure if there is no layer of dirt, fat, etc. on its surface. Major inclusions which were visible with a naked eye were treated as dirt. Up till now the term “steel purity” has been closely connected or even associated with steel impurities such as non-metallic inclusions. This is a major simplification as the term is much wider [1]. The degree of steel purity depends on: the content of harmful components (admixtures) in steel, the content, form, arrangement, size and type of non-metallic inclusions, chemical and phase heterogeneity (segregation) of metal [2]. The term “pure steel” has been changing its meaning. It had a different meaning for open-hearth steel, different one for electric steel produced in the 19th century, for steel melted 20-30 years ago and even a different one for modern steel. The understanding of this term changes with the development of steel melting and refining technology. It can also have a different meaning depending on the materials used in steelmaking. The requirements of recipients are also a factor affecting “the pure steel” criteria [1].

The oxygen content in steel in approximation is directly proportional to the amount of oxide inclusions. It is similar when it comes to the nitrogen content. Most often the nitrogen content in steel increases as a result of the contact between liquid metal and the atmospheric air. What follows is an increase in the oxygen content and as a result an increase of non-metallic oxide inclusions [1].

The total content of oxygen in steel is the sum of the dissolved oxygen (free) and oxygen bound in oxides (non-metallic inclusions). Free oxygen or active oxygen in liquid ferroalloys can be measured. The content of total oxygen is controlled by thermo-dynamic equilibrium with deoxidizing elements, e.g. aluminium [1]. The differences between the nitrogen content in steel when it comes to subsequent stages of secondary metallurgy (e.g. ladle and intermediate ladle) suggest secondary oxidation between subsequent operations. Steel deoxidation (especially low carbon steel deoxidation) creates favourable conditions for air absorption. The nitrogen content indirectly influences the level of total oxygen in steel and as a result it influences not only steel purity but also possible quality problems due to inclusions from secondary oxidation [1]. Hydrogen is another important element which influences the quality of steel. It is important to control the hydrogen content in steel during the process as it has a harmful influence on the mechanical properties of ready-made products. Even several ppm of hydrogen in steel can cause the occurrence of flakes, blisters as well as a decrease in ductility and an increase in brittleness. The main sources of hydrogen include the humidity of the atmosphere which the liquid metal comes in contact with, the humidity of materials introduced into the bath, mostly ferroalloys and slag forming additives [1].

Currently the purity of steel tends to be considered from the point of view of chemical purity and metallurgical purity. The chemical purity of steel is connected to its chemical composition and the content of harmful and trace elements in steel. Metallurgical purity, on the other hand, is connected to the occurrence of non-metallic inclusions in steel, which affect negatively the steel treatment process and steel casting as well as the properties of final products. It is important to know if the inclusions are “hard” or “soft” in comparison to the metal matrix. Metallurgical purity is defined on the basis of the assessed amount of inclusions or the content of elements which the inclusions consist of [1].

To sum up, the term “pure steel” refers to such steel which contains the least of non-metallic inclusions (with the size of under 5 μm) and of harmful components for a particular type of steel. It is a homogenous constant not only when it comes to the decomposition of non-metallic inclusions, but also the chemical composition and crystallization state. The content of non-metallic inclusions in steel is only one of the factors which decide about steel purity. It is, however, closely connected to the other two factors. An increase in the content of non-metallic inclusions can be caused by an increase in the amount of harmful components. An increase in segregation is also very often connected to an increase in heterogeneity when it comes to the arrangement of non-metallic inclusions [1].

The technology of producing pure steel should be based on the following rules:

- oxygen dissolved in liquid steel during melting and refinement must be bound into solid or gaseous oxides and removed before steel casting,
- at each stage the sources of secondary oxidation of steel must be eliminated,
- when in contact with liquid steel refractory materials (ceramic) must be stable and resistant to corrosion and erosion,
- physical retention of liquid slag and powder in metal during the refining process and casting must be eliminated [1].

In order to improve the purity of steel certain precautions and methods preventing secondary oxidation of steel should be introduced during secondary metallurgy [3].

2. Production technologies used for obtaining ultra-pure steel

Typical markets for cast ingots include: the power industry (e.g. power plant shafts, turbine vanes); the oil and gas industry (seamless pipes); the aircraft industry (shafts, turbines, engine parts); medical engineering; ship construction (engine and drive shafts); machine construction and mechanical engineering (heavy forgings, bearings, drive rack wheels) as well as motor technology (shafts, axles). As far as the quality of ingots is concerned, the recipients require low segregation level and microscopic and macroscopic purity of the ingot. The segregations can be the result of the influence of temperature and casting rate, the form of ingots, the treatment of ingot head and the type of steel. In case of ingots intended for further treatment using rolling and forging equipment it is important to make sure that their surface is impeccable and the inner structure meets the requirements set for their future use. Some defects can lead to discarding the ingot. Typical surface defects of the ingot include transverse longitudinal cracks, deslagging, surface blisters and pores, peeling and a mosaic network of cracks.

For the past few years, especially in Europe but also in Asia, there has been an increasing demand for the electroslag melting process that could produce ingots with the mass between 100 and 250 Mg. Ingots of that size can be used mainly in the power industry. Pressure containers of large sizes can also be made of these ingots [5]. An Italian company Societadelle Fucine (SdF) produces very big shafts intended mostly for power plants and rolling mills. This company is capable of meeting high requirements set for the products used in the power sector. New generators for power plants can offer 1600 MW of electric energy. Due to this fact generators need to have high resistance. Apart from one competitor in Japan, SdF is the sole supplier in the world that meets such requirements thanks to installing an innovative VOD device. The company also produces containers for the petrochemical industry and the nuclear industry. Vapour turbines and generators are used by power plants all over the world and must meet the highest standards of quality [6].

The steelworks at BGH Edelstahl GmbH Deutschland have good results due to an approach based on adapting production to a particular branch or the real demand on the market. High efficiency arc furnace allows melting liquid steel from scrap metal, alloy additives and other additives in melting amounts from 35 to 42 Mg. The used devices guarantee not only an excellent quality of materials from the purity point of view but also the precision of chemical composition. They also allow an optimization of costs in case of special use materials. In Lippendorf BGH has a 7.5 Mg multi-chamber vacuum induction furnace which makes it possible to produce materials of highest purity. Melting high quality scrap metal and alloy additives in a vacuum as well as vacuum degassing allow the production of special types of high purity steel. The casting process is aimed at forming liquid steel into products which will be suitable as an entry material for further plastic treatment. Furthermore, thanks to casting technology it is possible to influence significantly the properties of semi-products and to control the crystal structure, homogeneity and purity of metal. Another way to improve the purity is to melt steel electrodes in devices used for conventional and vacuum electroslag melting of steel. This method not only allows to increase the degree of steel purity but also to influence the chemical composition of the melt and the related improvement of material properties [6].

Kia Steel Co., Ltd., Kusan City in South Korea produces high purity steel (that means that the oxygen content is below 10 ppm) using two electric arc furnaces with the capacity of 60 Mg, two ladle furnaces with the same capacity as the electric ones as well as VD and VOD devices. They cast steel using a bottom pouring system. ESAC–Evergreen Superior Alloys Corp., HsinYing in Taiwan uses for the same purpose a 25 Mg electric and ladle furnace, a VD device and steel is cast into ingots using a bottom pouring system, too [7].

The improvement of metallurgical purity can be guaranteed by decreasing the total oxygen content in steel. At Georgsmarienhütte steelworks research was conducted into the improvement of steel purity in the intermediate ladle and the steel-casting ladle. Closing the intermediate ladle in a protective atmosphere hinders the secondary oxidation of steel in such a way that total oxygen after 1 minute from the start of casting amounted to approx. 10 ppm. Without closing the intermediate ladle in a protective atmosphere it is possible to obtain such values only after 25 min of casting. It is also possible to improve the purity of steel by using appropriate precautions during each refining process and methods preventing the secondary oxidation of steel [2].

Introducing alloy additives in the form of ferroalloys into steel is one of the most important stages of secondary metallurgy. The influence of ferroalloy impurities on the purity of steel is a major concern. The influence of ferroalloy purity on the quality of steel is obvious as the ferroalloy additives are introduced at the final stage of steel melting. High total oxygen content in ferroalloys inevitably affects the purity of steel.

Refractory materials are one of the major sources of non-metallic inclusions in steel during refining steel in the ladle. While steel is cast into an ingot mould or an intermediate ladle, liquid steel is moving together with slag with which it is covered. Slag covers the walls of the ladle and solidifies after the ladle cools down.

In order to improve the purity of steel researchers investigated the issue of refining liquid steel during solidification in the ingot mould. To do so they drew on a patented device and a method which involves the use of heat produced during solidification and the use of the refining properties of slag. As far as the concepts of heating the steel in the hot top (using the heat produced during the solidification of the ingot body) and refining steel with appropriate slag, it is common to use physical and chemical properties of refining slag to absorb the non-metallic inclusions flowing with the streams of liquid steel, moving around in the ingot mould when the ingot is solidifying.

In order to ensure appropriate temperature conditions it is necessary to use an insulating layer during a period of time which in approximation amounts to a third of the foreseen solidification time. This creates favourable conditions for refining steel from non-metallic inclusions, which are produced during solidification, and from the most dangerous exogenous inclusions from secondary metallurgy – including especially inclusions coming from the erosion of refractory materials of the bottom pouring system [4].

3. Own research

3.1. Research methodology

The aim of this research is to analyze the production technology of ultra-pure steel intended for casting forging ingots. The investigated arc furnace has an eccentric bottom outlet opening. Its walls and roof are water-cooled. Melting scrap metal in the electric arc furnace involves adding carburizers, slag forming materials and ferroalloys with the use of gaseous oxygen. The refining process is conducted at the ladle furnace station.

After it is finished vacuum degassing of steel begins in the vacuum chamber. Steel degassing is conducted using the surface method by mixing the bath with argon. The next step consists in bottom pouring of steel into ingot moulds. Heating the excess sprue of the forging ingots begins after casting is over. Exothermic powder is used for all ingots (it is applied in portions). Additionally insulating powder is used. The final stage of the process involves transporting ingots in hot state for further platic treatment.

Data from 24 melts were analyzed using melting logs for chromium-nickel-molibdenum steel. Final concentration of oxygen was studied as it, among other factors, determines the purity of steel. The requirements for very high purity steel are as follows: the final concentration of oxygen dissolved in steel must amount to no more that 10 ppm. The activity measurements concerning oxygen in liquid steel are conducted using an actinometer produced by CELOX. These measurements are conducted after the refining process is finished. The final concentration of hydrogen and the content of sulphur in particular melts were also investigated. Authors also studied the quantitative content of non-metallic inclusions in the samples taken from forgings. These forgings were made of ingots cast using a bottom pouring system with conventional "ingot head" heating and with steel heating and refining in a hot top under slag made of insulating and refining powder.

The next stage of research involved theoretical simulations of production technologies of refining slag with the use of: lime and coal, bauxite and carbide. Simulations involved the use of melting logs. The conducted simulations were aimed at assessing the optimal share of bauxite in producing refining slag. The simulations were performed using thermodynamic software FactSage 5.5.

Equilib, which is one of the most important modules of FactSage, was used to conduct simulations concerning the production of refining slag. The module is responsible for the Gibbs energy minimization. In the course of conducting calculations it is necessary to provide the amount and chemical composition of reacting substances and the conditions in which the process takes place (temperature, pressure). The calculation results are presented in Results Window. Masses of paricular products correspond to the mass balance of the process as well as to the lowest possible value for free enthalpy in case of particular reaction products.

The possibilities that Equilib gives were used to make calculations in the course of the simulations concerning refining slag production. By changing the components, their share and the process temperature it was possible to assess the chemical composition of the obtained metallic and non-metallic phase in the equilibrium state.

3.2. Research results and their analysis

Final research results concerning the content of oxygen dissolved in steel, the content of sulphur and the final content of hydrogen after vacuum refinement were shown in Table 1.

On analysing final research results concerning the oxygen content in steel, it was concluded that even though the average oxygen content amounts only to 8.29 ppm (and is lower than required for these particular types of steel), still in several cases the final oxygen content was higher than allowed. A similar situation occurred when sulphur and hydrogen contents were analysed. The final average content was 30 ppm and was in accordance with the requirements, yet in 2 melts the final content of sulphur exceeded the admissible levels. Analysing the final concentration of hydrogen showed that maintaining its admissible concentration at the level of 0.8 ppm is not easy in the conditions of the investigated steelworks. Two analysed melts did not fulfil this requirement. It could be attributed to the depressurization of the vacuum during the final stage of tapping when metal goes through the hot top. One could also suggest that the analysed steelworks, which wants to fulfil the strict requirements, should maybe consider investing in a vacuum device, e.g. of VOD type.

Table 1.
Final measurement results of oxygen, sulphur and hydrogen content in the analyzed melts

Measurement parameter		Value, ppm
Oxygen	*average*	*8.29*
	max.	11.06
	min.	4.71
Sulphur	*average*	*30*
	max.	50
	min.	20
Hydrogen	*average*	*0.8*
	max.	1
	min.	0.7

Simulations based on melting logs were also performed. Apart from studying the influence of slag additives such as: CaO, CaC_2 or the refining mixture (which were used during the ladle refining process), research focused how the presence of bauxite or its lack affects the process. The simulations helped compare the share of non-metallic phase that was being produced.

The obtained results show that bauxite used as a slag forming material increases the share of the mass of liquid phase and what follows decreases the share of solid phase – solid precipitations, including lime.

The subsequent studies were aimed at assessing the optimal share of bauxite in the production of refining slag. The results of this part of simulation were presented in Table 2 and in Figure 1.

Table 2.
The share of solid and liquid phase in the produced slag

Simulation No.	Liquid phase share, %	Solid phase share, %
1.	21.01	78.99
2.	38.03	61.97
3.	50.94	49.06
4.	73.80	26.20
5.	83.28	16.72
6.	87.64	12.36
7.	91.81	8.19
8.	93.35	6.65
9.	93.67	6.33
10.	93.74	6.26

The conducted simulations allowed to asses the optimal share of bauxite in the production of refining slag. During the tenth simulation (Table 2) 93.74% of the liquid phase was obtained. It had the following chemical composition: 58.2% CaO, 37.97% Al_2O_3, 3.11% SiO_2, 0.67% MgO, 0.12% CaS. The chemical composition was complemented with such compounds as: FeO,

TiO_2, Ti_2O_3, MgS, FeS, Fe_2O_3 – yet their amounts were minimal. The obtained slag has a refining character.

Figure 1 presents the share of the undissolved lime in slag mass in relations with the bauxite share in slag forming materials used.

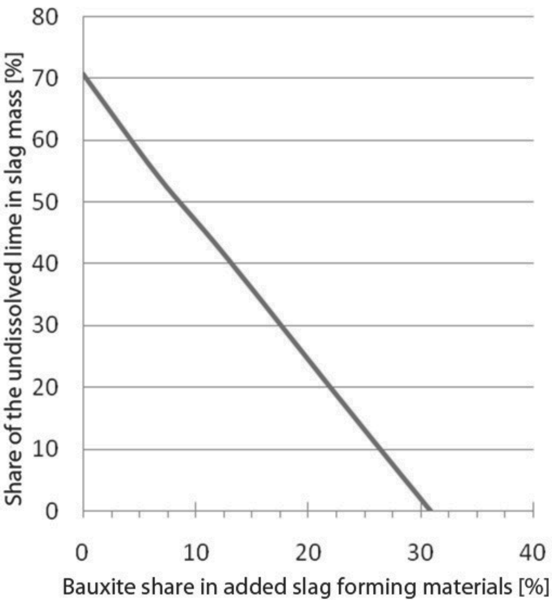

Fig. 1. The share of undissolved lime in slag mass in relation to the bauxite share in added slag forming materials

Increasing the bauxite share causes the liquid phase share to increase, too. It also leads to a more intense dissolution of lime. The lack of lime pieces in slag was observed when the share of bauxite amounted to 30.9%. The analyzed melting log shows that 50 g of bauxite was used, which was 6.7%. One could conclude that it is not enough to sustain the good quality of refining slag, which is crucial in terms of producing high quality steel. The remaining shares were as follows: 49.36% of lime, 4.94% of carbide and 14.81% of refining mixture. The simulations do not bring a ready made recipe but only suggest how the results could be improved. Steelworks save money when it comes to slag forming materials, which in turn affects the steel quality.

Table 3 presents quantitative measurement results concerning non-metallic inclusions in samples taken from investigated forgings. These forgings were made of ingots cast using a bottom pouring system with conventional heating of the "ingot head". They were also made of ingots cast with steel heating and refining in a hot top under slag produced from insulating and refining powder.

The data from Table 3 show that even steel refining under the heating layer improves the purity of steel – which is measured by analyzing the share of surface non-metallic inclusions. Steel refining process using an appropriately selected refining slag with basic character and with physical properties adapted to the temperature (which is close to liquidus temperature) can clearly decrease the amount of non-metallic inclusions and can decrease their size. It can aslo affect the shape of inclusions – making them almost spherical in shape, with the highest shape coefficient close to a unit.

Table 3.
Quantitative measurement results of oxides and sulfides in forgings samples made of ingots – heated in a conventional way (Technology A) or heated and refined under refining slag (Technology B) or under insulating powder (Technology C)

Measurement parameter		Technol. A	Technol. B	Technol. C
Surface inclusions share, %	*Average value*	*0.055*	*0.047*	*0.027*
	max.	0.154	0.080	0.039
	min.	0.017	0.013	0.020
Equivalent diameter, µm	*Average value*	*6.60*	*2.90*	*2.55*
	max.	11.19	4.64	2.33
	min.	3.16	2.56	2.89
Maximum diameter, µm	*Average value*	*9.01*	*4.99*	*4.75*
	max.	14.66	7.05	5.74
	min.	4.76	3.96	4.10
Minimal diameter, µm	*Average value*	*5.03*	*2.54*	*3.23*
	max.	8.70	3.15	3.70
	min.	2.69	1.93	3.00
Shape factor, min/max	*Average value*	*0.56*	*0.68*	*0.86*
	max.	0.71	0.87	0.88
	min.	0.43	0.60	0.84

This method should be used in particular for casting high mass ingots (both in case of the bottom pouring system and the vacuum system) in order to optimize the method and to continue further research connected to improving the purity of steel used to produce forgings for shafts in the power industry [4].

In Poland and abroad forging ingots (with the mass up to 80 Mg) are cast using the bottom pouring system after vacuum degassing of steel in a ladle or using RH, DH methods to obtain hydrogen content under 0.8 ppm. However, during casting the content of hydrogen increases in steel up to approx. 1.2-1.4 ppm, which may result in the appearance of certain defects in high mass ingots such as hydrogen flakes. It is possible to prevent this from happening by using time and energy consuming thermal treatment to remove hydrogen by diffusion from solid steel. Due to the inefficiency of such a technology foreign steelworks (Japan, the USA, Germany, France, the Czech Republic, Brazil, Great Britain, Russia, South Korea) use their own patented solutions to produce high mass ingots (above 50 Mg). These solutions take into consideration local technical possibilities of steelworks. The methods used involve double steel degassing as well as vacuum casting into ingot moulds in order to ensure the hydrogen content below 1 ppm.

4. Summary

Research conducted into final concentrations of oxygen, hydrogen and sulphur on the basis of data from melting logs and the amount of non-metallic inclusions shows that developing the production technology for ultra-pure steel is not an easy task.

Analysing the final concentration of oxygen dissolved in steel on the basis of measuring the oxygen activity in liquid steel makes it possible to conclude that approx. 90% of analysed types of steel qualifies as high purity steel. As far as analysing the final concentration of hydrogen dissolved in steel is concerned, the results are not as good. Hydrogen concentration in such types of steel cannot be higher than 0.8 ppm. Studying sulphur concentration in the obtained products confirmed that the production of high purity steel is not an easy task both in the Polish and foreign conditions.

The results obtained from simulating the production technologies of refining slag with the use of FactSage software and melting logs prove that the use of bauxite as a slag forming material leads to an increase in liquid phase share and consequently to a decrease in the solid phase share – solid precipitations, including lime. As a result of increasing the bauxite share one could observe that the liquid phase share increased in comparison to the amount of solid precipitations flowing on the slag surface. What is most important from the point of view of steel purity is the fact that slag has a refining character. The content of FeO and MnO oxides is lower than 1%. Carbon reduces oxides from original slag, thanks to which slag no longer has an oxidizing character. As a result, refining processes can be conducted in a proper way. The optimal share of bauxite in forming refining slag was also presented.

No-slag tapping is rather difficult to perform and that is why one needs to take into consideration the mass and chemical composition of furnace slag while choosing slag forming additives.

While forming refining slag it is necessary to control closely its composition. At this stage it is important for steel to have a certain chemical composition and properties before it is cast. Slag forming materials (which include lime, bauxite, powders containing aluminium, deoxidizers) should be, if possible, of high quality. If selected carefully, they will form ladle slag which can ensure the production of high quality steel.

On the basis of the above mentioned considerations it seems reasonable to conclude that all stages of high purity steel production must be conducted conscientiously and meticulously. It is also vital to improve technological conditions of vacuum refinement of steel by equipping steelworks with new vacuum devices, e.g of VOD type.

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