

# HOT ISOSTATIC PRESSING (HIP): THE STATE OF THE ART & IMPROVEMENT ON TWO STEELS

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## Abstract

The present study is comprehensive of a brief critical review that deals with the effects related to the application of the Hot Isostatic Pressing (HIP) in a gas atmosphere and the fundamentals about its action on the material structure and its properties. The HIP can be profitably used to improve the strength and the toughness of the mechanical parts, especially when they are produced by a near net shape process. The foundry is certainly the most ancient near net shape process and it is still one of the most used to produce some parts for the automotive industry. The cast parts often show some chemical and structural disomogeneity, i.e. segregation and porosity, that are very detrimental for the mechanical performances of the material. The experimental task focused on the application of HIP treatment to two different cast steel, have pointed out the significant improvement of the mechanical properties and more homogeneous performances.

## Riassunto

Il presente studio presenta una breve introduzione critica riguardante gli effetti del metodo di pressatura isostatica a caldo in atmosfera gassosa e gli aspetti fisici fondamentali circa la sua azione sulla struttura del materiale e le sue proprietà. Il processo di pressatura a caldo può essere vantaggiosamente utilizzato per il miglioramento delle caratteristiche meccaniche di componenti che al termine del processo stesso abbiano una forma del tutto prossima al componente finale (near net shape process). La fonderia, tra questi processi, è certo quello di origine più antica ed è tuttora largamente impiegato per la produzione di componenti destinati all'impiego nell'industria automobilistica. Spesso i getti presentano caratteristiche di disomogeneità chimiche e strutturali, quali le porosità, che deteriorano le caratteristiche meccaniche del materiale. Gli effetti di questi difetti possono essere in parte rimossi proprio dall'HIP che, come dimostrano i test meccanici svolti su provini provenienti da getti realizzati su due differenti di tipi di acciaio, è in grado di produrre un incremento delle proprietà meccaniche ed una maggiore uniformità delle sue prestazioni.

## THE HIP PROCESS: TECHNOLOGY, AIMS AND CONTROLLING PARAMETERS

The hot isostatic pressing (HIP) is a process for the densification of the castings and of the powder metallurgy products. The mechanical performances of these products depend on the distribution, morphology and volume fraction of porosity having a detrimental effect on properties like fracture toughness, fatigue resistance and tensile strength [1]. The porosity of the castings is caused by shrinkage of the metal and the decrease of the gas solubility during the solidification and cooling steps, whereas in the powder metallurgy the porosity is present from the beginning of the sintering process as the holes between the powder particles. The basic function of a hot isostatic press (fig.1) is "to uniformly heat a given workload, while gas pressure is applied to all surfaces, with accurate control of temperature and pressure."

The pressure medium is a gas, such as argon, nitrogen, helium or air. A variety of heating elements can be used for the different temperature ranges. Thermocouples provide precise control of the temperature of both the heating elements and the workload.

The furnaces are of various types, to suit particular requirements:

1. Oxygen resistant kanthal heating elements for temperatures up to 1200°C. These allow hot loading and unloading of the workpieces.
2. Molybdenum heating elements for temperatures up to 1450°C; used mainly for densification of materials sensitive to surface contamination.

3. Graphite heating elements for temperatures of 2000°C or above, permit treatment of materials in either an argon or nitrogen atmosphere.

The heat shield assembly provides thermal insulation of the pressure vessel, and controls the conduction, convection and radiation of the internal gas.

A compressor system provides the required gas pressure. Gas can be obtained from bottles or from a cryogenic liquid system, and generally more than 90% of the gas can be recovered for re-use. The control systems range from manual to fully automatic, with varying degrees of sophistication, from simple analogue to fully integrated computer systems.

The cooling system keeps the temperature of the pressure vessel wall and covers at the required level. Cooling water is circulated through a heat exchanger in a closed re-cycle system (thus reducing water consumption).

The pressure vessel is the most important part of the hot isostatic press and can be built, for industrial applications, with:

- inside diameters up to 2000 mm or more

- pressures: from vacuum to 3000 bar
- temperatures: from 500°C to 2200°C (932°F to 4000°F)

The micro-porosity is one of the most important causes of fatigue crack initiation as well as non-metallic compounds that can form by the precipitation of oxide and/or nitride inclusions. The non-metallic inclusions cannot be eliminated after the solidification is completed, but micro-voids can be welded by means of strengths applied at high temperature. The HIP process aims at healing the voids that turn back from the shrinkage and gas evolution applying an isostatic pressure that cannot modify heterogeneously the shape of the treated products. The HIP is also used to relax the tensions within some metal matrix composites reinforced by ceramic particles or fibres, in which during the solidification some residual tensile strength are left at the interface between the metal matrix and the ceramic counterparts because of their different thermal expansion coefficient [2].

The technological parameters that should be controlled are: the temperature, the pressure applied, the timing of the different periods of tempera-

tures and strength application, because the HIP has to be performed by keeping great attention about the features of the dynamic behaviour of the processed materials, in which the relation between deformation speed and strength varies as an exponential function. It is worth noting that the permanence of the materials at high temperature can imply the coalescence of the grains of the hot metal that sometimes requires a successive strengthening treatment (i.e. aging treatment).

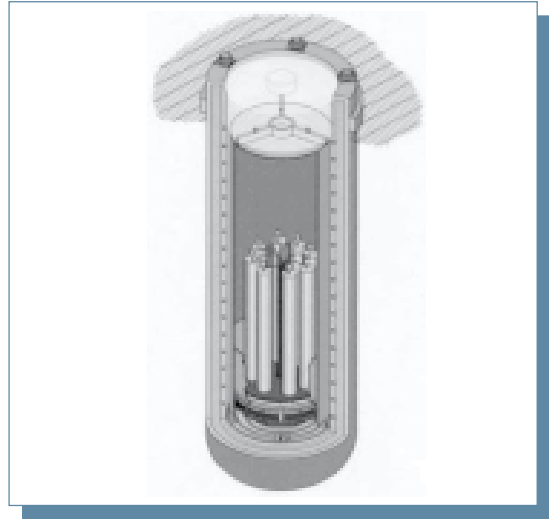


Fig. 1: Layout of a hot isostatic press

## THE DYNAMIC MATERIAL BEHAVIOUR

A classical approach based on the dynamic material model was firstly proposed by Gegel [3] for dense materials, as the cast products can be considered. But a more general formulation that includes the materials with a high volume fraction of voids was developed by Zhang et al. [4] to describe the phenomenon leading to the densification in the sintered products. The basis of this model is still the relation between the various mechanisms governing deformation at high temperature and the dissipative structure theory. The input deformation power is divided into two components:

- plastic deformation energy ( $G$ ), that is irreversibly dissipated during the material deformation;
- potential energy ( $J$ ) that is stored in the material by the microstructure evolution.

These two energy components can be supposed of this form:

$$G = \int \sigma_R d\varepsilon_R$$

$$J = \int \dot{\varepsilon}_R d\sigma_R$$

where  $\sigma_R$  and  $\dot{\varepsilon}_R$  are the flow stress and the corresponding flow strain rate for the materials respectively (fig.2).

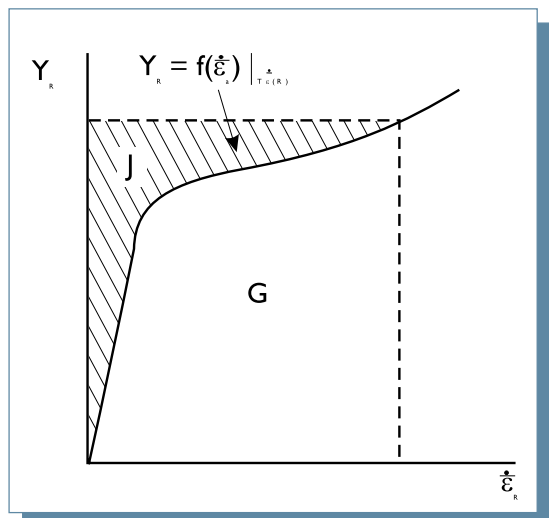


Fig. 2: An example of the flow stress and the corresponding flow strain rate

The HIP does not require the presence of any dissipation of energy by a working tool, but this last is performed by the workpiece isostatically pressured by the hot gas atmosphere. The instantaneous response of the material under pressure can be well interpolated by:

$$\sigma = K\dot{\epsilon}^m \Big|_T$$

where  $\sigma$  and  $\dot{\epsilon}$  are the real strength and the rate of deformation respectively,  $K$  and  $m$  are two characteristic constants of the material that have to be determined by experimental tasks. However,  $m$  can be considered constant only for pure metals, but in the most of the engineering alloys varies as function of temperature and strain rate.

The deformation process has not to lead to the instability of the plastic flow of the materials, but it aims at maximizing the dissipation of the energy governing the beneficial microstructure transformation. So, it is possible to define a criterion for the dissipative efficiency:

$$\eta = \frac{J}{J_{\max}} = \frac{m}{m+1}$$

$$J_{\max} = \frac{\sigma\dot{\epsilon}}{2}$$

The combination of  $T$ ,  $\dot{\epsilon}$  that gives the highest value of the dissipative coefficient has to be searched for, but attention has to be kept to avoid the

## DENSIFICATION, CONSOLIDATION MECHANISMS AND RESIDUAL STRESSES RELAXATION

The mechanisms of densification involved in the cast products are relatively simpler than those involved in the densification of metal powder or metal matrix composite reinforced by ceramic particles. The densification of cast alloys is well described by the dynamic materials model approach and the use of a steady-state creep rates law as the constitutive relation:

$$\dot{\epsilon}_s = A\sigma^{n_a} \exp(-Q_a / R_g T)$$

where  $Q_a$  is the activation energy for the creep process,  $R_g$  is the gas constant,  $A$  and  $n_a$  are characteristic constants of the materials.

For the densification of metal powder products a complete understanding of the phenomena that occurred makes necessary the evaluation of other constitutive relations ruling the process and the friction phenomena caused by the relative movement of the particles.

Some experiments using transparent metal type compounds such camphene and succinotrile [5] were performed to study the evolution of the densification in the HIP of the powders. The plastic yielding of the begin-

ning of damage mechanisms, but in the case of HIP (characterized by isostatic compression state) the formation of cavities and/or micro-cracks is not probable, so that it is possible to suppose that the whole energy is spent in metallurgical process such as the closure of micro-voids and porosity or other phenomena of little importance in the HIP process (i.e. dynamic recovery, phase transformation, recrystallization).

In the case of materials with a high void volume fraction, as the powder metallurgy products before the densification, the schema proposed has to be modified including the effect of the variation of the density that influences the  $\sigma_R$  and values  $\dot{\epsilon}_R$ . So the dissipative efficiency coefficient

$$\eta = 2 \left\{ 1 - \frac{[R(1-R_c)/(R-R_c)]^{m+1}}{m+1} \right\}$$

where  $R$  and  $R_c$  are the density and then relative density of the materials respectively. This last formulation of the dynamic material model approach is strictly needed for the porous materials, but it can be avoided for the castings.

ning steps is followed by time dependent mechanisms of power law creep and the following diffusion phenomena.

The standard HIP law assumes steady-state creep rates, but a more detailed approach implies the inclusion of the friction stress among the different particles. A reliable description of friction behaviour can be made by the hypothesis of a threshold value ( $\sigma_0$ ) and the linear dependency of friction stress ( $\sigma_b$ ) on the effective applied one ( $\sigma$ ):

$$\sigma_b = K_b \sigma + \sigma_0$$

where  $K_b$  is the constant of linear dependency. The power law should be modified in the form:

$$\dot{\epsilon}_s = C(\sigma + \sigma_b)^{n_b} \exp(-Q_b/R_g T)$$

and  $C$ ,  $n_b$ ,  $Q_b$  can often be approximated by  $A$ ,  $n_a$ ,  $Q_a$  respectively [6].

During the first stages of densification the internal stresses of the pressurized structure are higher than the applied pressure, because the internal volume is characterized by an amount of micro-

voids that decreases through the process. The increase of the relative density of the pressurized structure causes an increase of the resistant area and a related decrease of the effective stress acting within the workpiece. The friction begins to be an important factor when the effective stress becomes small, then this aspect is significant only after the first stages of densification, then the last model that includes the scaling between the friction and the applied stresses showed a good agreement with the experimental data. Thus, the retarding effect of friction resistance on the creep deformation is lowered for the decrease of the effective stress after the increase of the resistant area caused by the densification itself.

The diffusion phenomena can be summarized and classified as boundary diffusion, volume diffusion and diffusion flow [7,8,9,10,11,12,13,14]. On the other hand, it is still worth noting the direct relation between the kinetics of diffusion with the effective applied stresses and this fact means a variation in the kinetics of diffusion processes among the different steps of densification. The decrease of the effective internal stress lowers the diffusion kinetics.

But other researches [15] about the metal powder consolidation point out the importance of other phenomena influencing this process. First

of all the relative movement of the particles has a significant contribute to early stages of densification and this motion is closely related to the particle sizes. In the absence of microstructural differences among the particles the first deformations concentrate in the smaller particles at the contact between the small particles themselves and the big ones, so they can help the rigid body motion of the largest particles. During the first stages of consolidation the coordination number distributions shift to higher coordination number with a simultaneous deformation and shape change of the particles. As the densification proceeds, the modification of the packing takes place and the higher coordination configuration becomes stable.

The HIP can have a great role in processes aimed at relaxing the local residual stresses left in the metal matrix composite after the cooling. Dvorak et al. [16] studied this phenomenon using a SiC/Ti composite, in which the reinforcing SiC fibers shows an elastic behaviour and the metal matrix a visco-elastic one.

The source of the residual stresses is the different thermal expansion coefficient belonging to the ceramic and metal matrix materials. The internal stresses states are modified significantly by the variation of processing parameters of HIP during the cooling of the composite. The relaxation relies on the visco-elastic behaviour of the metal matrix characterized by straining during the application of hydrostatic pressure. Most of the plastic straining is accomplished at the low yield strength that can be reached by a high temperature. The high confining pressure and low cooling rates are also helpful in the relaxation process. This relations are not only typical of the composite, but the same results can be obtained in the treatment of spray cast and diffusion bonded system subjected to the same thermo-mechanical process.

## MATERIALS, EXPERIMENTAL PROCEDURE AND RESULTS

The experimental procedure has been performed on two different types of steels (table I):

- the first one for quenching and tempering treatment;
- and the second one aimed at carburising treatment.

The two steels have been produced in a vacuum system and then melted again to produce under vacuum castings of cylindrical shape with 100mm diameter and 170mm high. Fifty samples of each steel have been tested, but only on one half of these the HIP has been performed and this procedure is ruled according to the following parameters:

- initial temperature 50°C at 30MPa;
- reheating from the initial temperature at 1250°C with the pressure that varies between 30 to 100MPa in 8400s;
- permanence at 1250°C and 100MPa for 4200s;
- air-cooling to the environment temperature performed in 5000s.

The samples of steel 1 and steel 2 have been sub-

TABLE I. CHEMICAL COMPOSITION OF THE ANALYSED STEEL

	%C	%Mn	%Si	%Al	%Cr	%Ni	%V	%Cu
Steel 1	0.38	0.58	0.21	0.004	0.85	1.1	-	0.1
Steel 2	0.19	1	0.23	0.002	1.3	0.89	0.02	0.22

jected to two different preparation procedures. The steel 1 undergoes:

- re-heating at 880°C and permanence at this temperature for 2400 s;
- oil quenching;
- tempering at 550°C for 7200s;
- final cooling in air;

while the steel 2 is so treated:

- on the sample of these steels the carburising process has been performed at 920°C for 1800s;
- then these samples have been cooled and subjected to a quenching and tempering treatment with same parameter of the steel 1 samples.

The treated bar undergoes a metallographic analyses devoted to point out the presence of micro-porosity due to the shrinkage of solidification. Tensile tests to determine the ultimate stress, yield stress, percentage elongation and the percentage reduction of the area have been performed. Charpy toughness tests have been implemented on KV samples to determine the eventual increase of the toughness feature.

TABLE II.

Steel 1	R (MPa)	R <sub>p0.2</sub> (MPa)	%A	%Z	KV (J/m <sup>2</sup> )
Average	1028	931	10.8	25.3	51.5
St.dev.	10.6	8.4	11.1	6.4	8.2
Steel 1 (HIP)	R (MPa)	R <sub>p0.2</sub> (MPa)	%A	%Z	KV (J/m <sup>2</sup> )
Average	1034.2	950.1	17.2	54.1	69.4
St.dev.	4.1	4.2	5.5	2.8	4.2

TABLE III.

Steel 2	R (MPa)	R <sub>p0.2</sub> (MPa)	%A	%Z	KV (J/m <sup>2</sup> )
Average	1350	1095	4.8	17.9	56.5
St.dev.	50.6	70.8	2.1	4.5	10.1
Steel 2 (HIP)	R (MPa)	R <sub>p0.2</sub> (MPa)	%A	%Z	KV (J/m <sup>2</sup> )
Average	1435	1161	9.1	31.7	73.6
St.dev.	7.6	5.5	0.7	1.1	5.4

## DISCUSSION

A qualitative metallographic evaluation of the samples indicates an important decrease of the micro-porosity examples in HIP treated steels, evidencing that a densification of the steels occurred. It is worth noting a general increase of all the mechanical properties of the steel samples belonging to the castings that have been subjected to the HIP process. Steel 1 shows a clearer increase of the fracture and yield stress than steel 2. These mechanical results and the qualitative metallographic analysis seem to confirm that some detrimental effects that could take place due to the permanence at high temperature, i.e. coarser grain distribution, do not occur, otherwise a simultaneous lowering effect of the tensile and toughness features would have happened.

Moreover, the results point out a significant decrease of the dispersion of all the values around the average one. This fact is extremely important because it indicates that the mechanical features of the isostatic-pressured steels show the reduction of the heterogeneity within the cast material alloys and this fact makes the designing task less critical and more reliable. This phenomenon implies more reliability of the material in the design of the components aimed at the structural applications.

Another interesting result concerns the simultaneous improvement of the tensile characteristics and ductility and toughness ones, which is an important task in the reinforcement procedure for steels of structural application.

However, it has to be pointed out that the significant increasing of the mechanical features that has been observed could also be due to the fact that the previous casting process has been developed under the vacuum conditions so that the initial casting probably shows a better structure than that which can be obtained by traditional methods in not controlled atmosphere. On the other hand, it is an appreciable result that HIP revealed to be a method able to reach a significant improvement in the castings even produced by the more sophisticated methods, so that in the critical application of the components produced by the foundry techniques it can become a non-negligible step of the productive process.

The results of this task of the mechanical tests give the results shown in table II and in table III.

## CONCLUSIONS

The study points out the beneficial effect of the HIP process applied to the cast products even when applied to the casting products obtained by yet careful process, as the casting under vacuum condition that is performed in this study.

An improvement of the tensile and toughness features of the two investigated steels for special structural applications is evidenced.

The more homogeneous structure of the cast product is indicated by the significant reduction of the standard deviation from the average value of the main mechanical characteristics. This result permits more reliable designing task.

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