

Organic Impregnation System

Introduction

In general, to provide new functions to the porous object and to reinforce the original functions by infusing or osmosing liquid such as resins or solutions into holes of the porous object is called impregnation.

For familiar examples, there are furniture, that is free from bugs because the timber is impregnated with repellent and cloth provided with the function of flame resistance or non-wettability etc. by impregnating fire retardant or water-repellent.

Moreover, it is also possible to impregnate such as concrete and slates with resin to eliminate the defect of "brittleness" and to transform them to strong materials.

There are already products as well in our products, which are provided with functions by impregnating treatment. Three-sheet gasket is provided with the functions of both solid gaskets and liquid gaskets by impregnating nonwoven fabric or paper etc. with sealant and liquid gaskets. In the meantime, ThreeBond 1873 and 1874 are a sheet or a bag of nonwoven fabric, that was impregnated with volatile rust inhibitor solution to obtain corrosion protection.

In this technical news, we introduce organic impregnation. It is the method to osmose impregnating sealant, of which the main component is methacrylic acid ester monomer, into holes of the porous objects of casting products such as sintered metal, cast metal, and die-cast and to cure them in holes. By this organic impregnation, functions such as prevention of leakage, improvement in strength, and improvement in processing accuracy of the surface of sintered metal or casting products, and prevention of swell of coating films after the coating treatment are provided.

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1. Porosity of Cast Metal

Many people might wonder at our description that the casting products are porous objects. It is because they remind us the fine metal surface with unique luster; besides, we often see such a metal surface in fact.

There are few holes in metal products with press working or strip processing, made from steel plates or aluminum plates. However, it is because in casting products of cast metal or die-cast, holes will be generated in manufacturing anyway.

Casting products are manufactured by flowing or injecting melted metal (called “molten bath” in general) into molds previously made with sand or metal.

At this time, if impurities (oxides, flux, and water etc.) are mixed in the molten bath, holes called “gas holes” or “pinholes” are generated in casting products because of the gas generated from impurities due to high temperature. Holes are generated by impurities not only in “molten bath” but also in the surface or inside the mold, which is flowed into.

Furthermore, when metal coagulates from the melted liquid state to the cooled solid state, “shrinkage” always occurs. With this volume shrinkage, holes called “cracks” or “shrinkage cavities” are generated. That is the reason why we describe that casting products are porous objects.

In this technical news, we collectively call these holes “porosities”.

With pressure-resistance casting products, porosities will cause defects such as leakage or strength poverty. Therefore, several measures are devised at casting to prevent generation of porosities in products. For example, following measures are taken: to prepare ventholes in molds, to keep the “molten bath” to the proper temperature, to prepare a “gate riser”, a “pouring gate”, or a “chiller” to prevent volume shrinkage, and to consider radial thickness, shape, the number of the impression, and the layout at the design phase of the products etc.

Since the organic impregnation is to eliminate porosities by embedding inside porosities with resin, it is said that it is one of the porosity prevention measures.

Examples of Porosities ⁽¹⁾

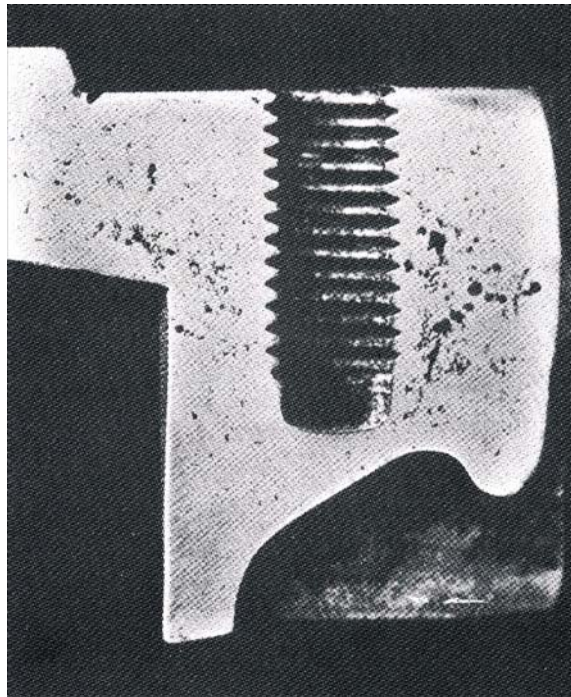


Photo 1. Defect B111-5a (Aluminum alloy, greensand mold)
Aluminum alloy cast metal (Si 6%, Cu 4%). There are “blowholes.” It was caused by outgassing defect of molten bath and wrong casting plan of the gate or the gate riser.

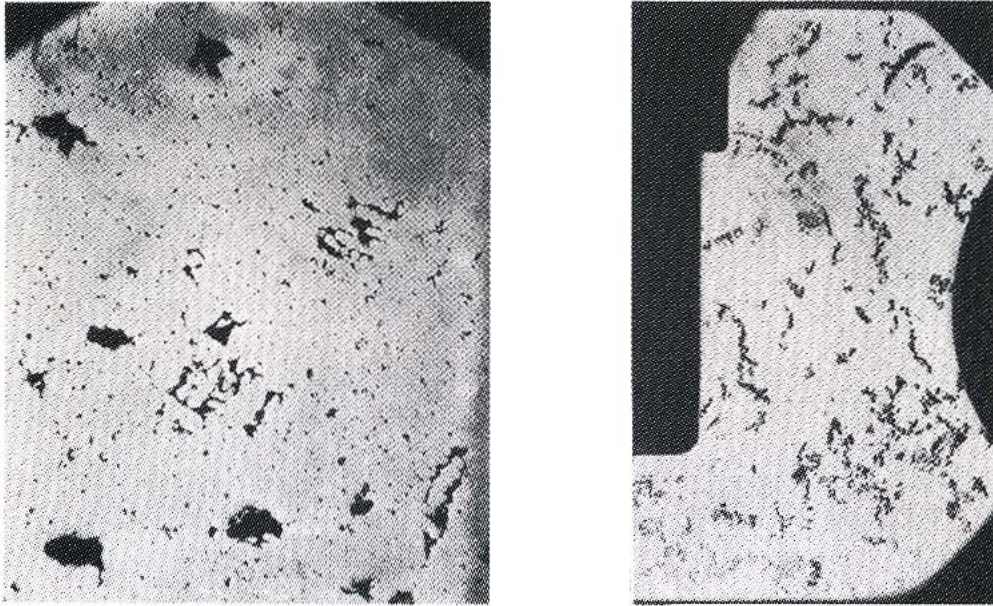


Photo 2. Defect B311-6 (Copper alloy)
“Microporosities” generated on the rim parts of bronze casting. Without etching.

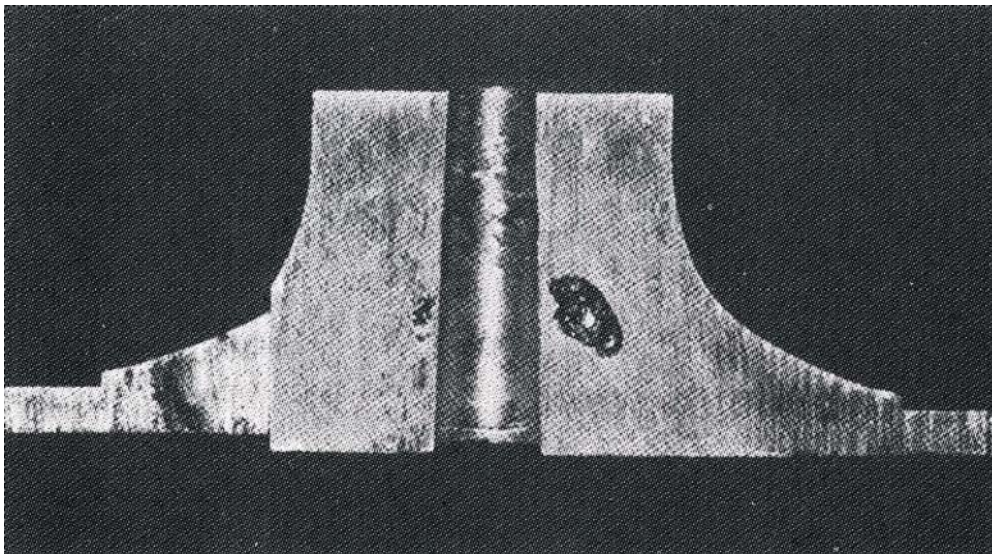


Photo 3. Defect B221-5h (Aluminum alloy, green sand mold)
Aluminum alloy (Si 12%) die casting. 170 mm ϕ , height 65 mm, weight 1.4kg. It was infused by the horn gate from downside. Because of the too much thickness, “shrinkage cavities” were generated. Countermeasures are as follows: to change the design, to use another alloy experimentally, and to change the temperature of the die.

2. Types of Impregnation Method

Although, casting products such as cast metal and die-cast are made not to contain porosities by various measures mentioned above, they cannot be prevented completely even with them.

In the meantime, even in case the porosities exist, there are almost no example as extremely large as the one

shown in the photo. With actual porosities, it is usually very difficult to find porosities with naked eyes after cutting. Even with small porosities like these, when pressure is applied, harmful effects such as leakage of liquid or gas occurs.

There are measures to impregnate these small porosities as follows:

Examples of Impregnation Method

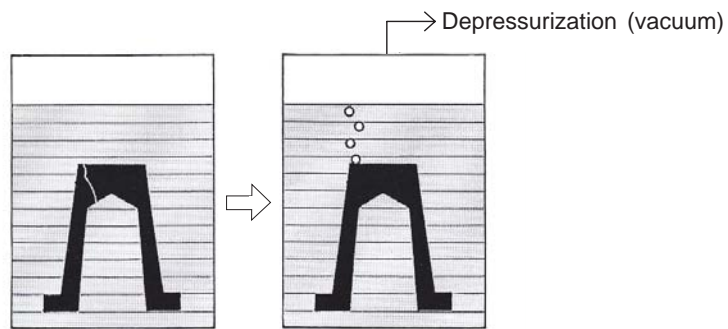


Figure 1. Dip-vacuum method

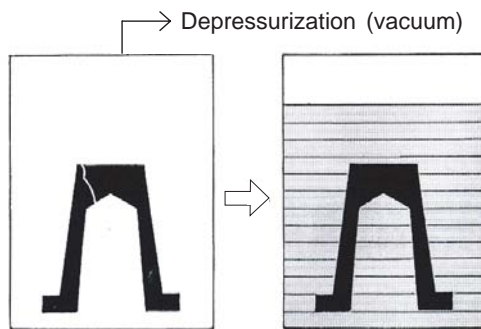


Figure 2. Vacuum-dip method

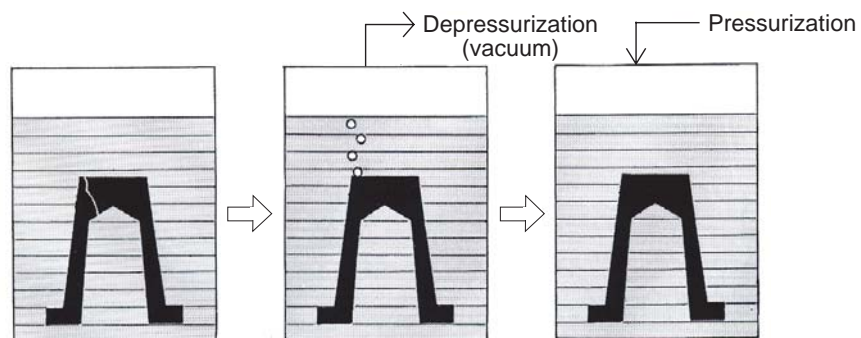


Figure 3. Dip-vacuum-pressure method

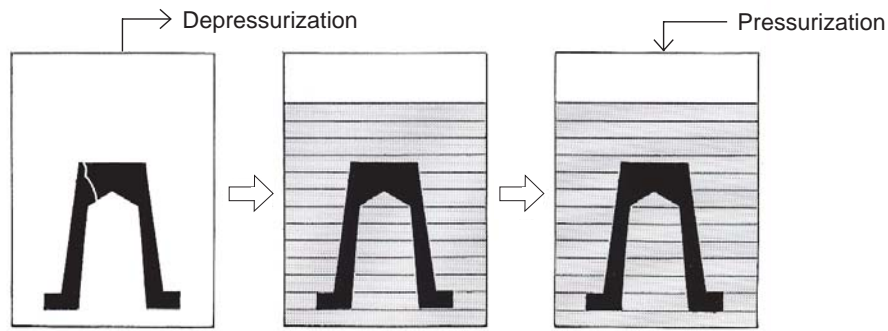


Figure 4. Vacuum-dip-pressure method

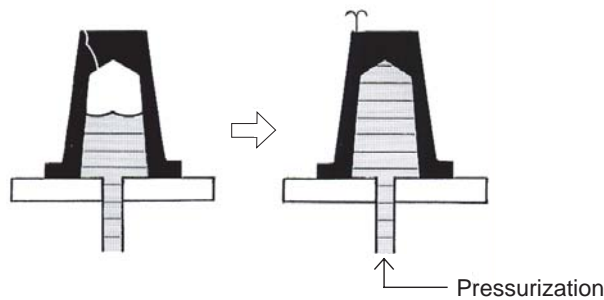


Figure 5. Internal-pressure method

The operation of impregnation uses the capillary phenomena. The solution, which osmose easily by capillary effect, has the following characteristics:

- ① Low viscosity.
- ② Highly wettable (low contact angle).
- ③ High surface tension.

The rapidity of osmosis of the solution is expressed as follows:

$$t = \frac{2\eta \cdot \ell^2}{r \cdot \gamma \cdot \cos \theta} \quad \text{(Equation-1)}$$

t : Time η : Viscosity ℓ : Depth of osmosis
 r : Diameter of the hole γ : Surface tension
 θ : Contact angle

However, besides osmosis due to capillary phenomena, substitution of air inside porosities to inflowing solution must be considered. When air is substituted with solution, since air will ascend as air bubbles, air is exhausted from porosities. If we suppose that, Stokes' equation can be applied.

$$u = 2\rho r^2 g / 9\eta \quad \text{(Equation-2)}^{(3)}$$

u : Ascension rate of air bubble
 ρ : Density difference between air and solution
 r : Radius of air bubble η : Viscosity
 g : Gravitational constant

If we suppose, $\rho \cong 1$, $r = 0.001$ cm, $\eta = 10$ cP, and $g = 980$ cm/s², the ascending speed of air bubbles will be $u \cong 2 \times 10^{-5}$ cm/sec, that means almost stationary state in appearance. Thus air will not be exhausted. Furthermore, since the air in the porosities is in the state of adsorption, more energy is required for exhaustion.

That is the reason to conduct vacuum process to degas the air and to let the solution to osmose into porosities. In case of high viscosity solution, it requires time to osmose into porosities as shown in (Equation 1). Therefore, press fit is conducted at pressurization process.

Regarding these impregnation method, vacuum-dip-pressure method and internal pressure method is defined by the United States Military Standard MIL-STD-276.

Methacrylic acid ester monomer-based impregnating sealant is excellent at osmotic property with the low viscosity of about 10cP, good wettability with the contact angle of 10 degrees, and the surface tension of about 35dyn/cm. Due to this, impregnation effect equivalent to vacuum-dip-pressure method is available even with the dip-vacuum method or vacuum-dip method with pressurization process omitted.

3. Types of Impregnating Sealant

With impregnation of casting products, water glass-based inorganic impregnation has been conducted from before. However, with inorganic impregnation, since the remaining ratio after heating is as low as 20 to 40% in general, it will shrink during curing. In addition, the “blowing” phenomenon, flowing out of impregnating sealant, which has been impregnated in porosities, while curing occurs. From these reasons, the yield of successful impregnation in case of impregnating treatment of casting products is generally around 50 to 80%.

Because of this low yield, impregnating treatment method is transitioned from inorganic impregnation to organic impregnation.

For impregnating sealant of organic impregnation, there are three types in general as follows: polyester type, anaerobic acrylic type, and heat curing acrylic type.

Table 1 shows the comparison of these impregnating sealants.

There is the United States Military Standard MIL-I-6869 for impregnating sealant as well, that defines performance such as sealing property, chemical resistance, and heat cycle resistance of impregnating sealants.

Table 1. Comparison of impregnating sealants ⁽⁴⁾

	Water glass	Polyester	Anaerobic acrylic	Heat curing acrylic
Main component	Sodium silicate	Diallyl phthalate/ Styrene	Methacrylic acid ester	Methacrylic acid ester
Curing mechanism	Disintegration of water and chemical reaction with carbonic acid gas	Thermal polymerization (45 min. in oil bath)	Anaerobic curing (4 to 6 hr. with room temperature curing)	Thermal polymerization (10 to 15 min. in 90 to 100°C water)
Remaining ratio after heating	About 20 to 40%	About 90 to 100%	About 90 to 100%	About 90 to 100%
Blowing	Exist	Exist	None	None
Cost [Compared with the value 1 of water glass]	1	1 – 2	8 to 10 times	8 to 10 times
Cleaning method	Water	Organic solvent or surfactant solution	Organic solvent or surfactant solution	Water
Wastewater treatment	Neutralizing processing is available	Wastewater treatment facility is needed	Wastewater treatment facility is needed	Wastewater treatment facility is needed
U.S. Military Standard MIL-I-6869	Not Available	Passed	Passed	Passed
Maintenance of impregnating solution	Easy	Easy	Check is needed once per day Low temperature and oxygen supply are required during storage	If low temperature storage is conducted, maintenance of solution is easy
Remarks	Yield is as low as 50 to 80%	Since styrene is contained, local exhaustion and fire precautions are needed	Maintenance of solution is costly	Aluminum is sometimes discolored during curing

4. ThreeBond 3932 Series

shows the comparison of these three types and Table 3 shows the characteristics of ThreeBond 3932.

Our organic impregnating sealants are heat curing acrylic type, with which following 3 types are available. Table 2

Table 2. Types of ThreeBond 3932 Series (Organic Impregnating Sealant)

	ThreeBond 3932	ThreeBond 3932 C	ThreeBond 3932 L
Appearance	Colorless	Colorless	Colorless
Specific gravity	1.07	1.07	1.02
Viscosity	10cP	12cP	12cP
Compounding ratio (Main agent/curing agent)	9/1	9/1	100/2
Remarks	General purpose type	Type with high detergency For small cast metal	Type provided with anaerobic curing

Table 3. Characteristics of ThreeBond 3932

Test items		Conditions	Characteristics	Test method
Compounding ratio		(Main agent/curing agent)	9:1	Weight ratio
Flash point (°C)		Main agent	110	JIS K 2265
		Curing agent	150	JIS K 2265
Surface tension (dyn/cm)		Mixture	35.5	3TS *
Hydrogen ion concentration (pH)		Mixture	4 – 6	3TS
Remaining ratio after heating (%)		Mixture	91	3TS
Polymerization starting temperature (°C)		Mixture	84	3TS
Hardness of cured material		Shore D	70 – 82	3TS
Thermal decomposition starting temperature (heat resistance) (°C)		Cured material	200	3TS
Sealing	Sintered component	Test media (air pressure)	2kgf/cm ² pass	3TS
	Oil pump	Test media (hydraulic pressure)	400kgf/cm ² pass	Real product
Solution resistance (%) 7 days of dip	Fuel (gasoline)	45 – 50°C	+0.4	3TS Rate of weight change of cured material
	Fuel (diesel)	45 – 50°C	+1.1	
	Lubricant (motor oil)	100°C	-1.8	
	Lubricant (gear oil)	100°C	+0.8	
	Water	95 – 100°C	+8.2	
	LPG	Room temperature	+1.3	
	Freon gas	Room temperature	+1.9	
	Solvent toluene	Room temperature	+1.3	
	Acid HCl (pH1)	Room temperature	+14.3	
Alkali NaOH (pH13)	Room temperature	+15.8		

- 3TS is the abbreviation of the ThreeBond Standard Test method.
- These data are measured values and not standard values.
- Since the examination method is not standardized in Japan, there are many cases where the followings are quoted or examined:
 - 1) Examination with real products and achievements
 - 2) Cured material of impregnant itself
 - 3) MIL-I-6869 D Standard
 - 4) Use of sintered pairs or capillaries.

5. Organic Impregnation Process

Our standard process of vacuum-dip-pressure method of organic impregnation is shown in Table 4. One cycle of impregnating treatment is generally about 60 minutes.

6. Impregnating Apparatus

Our standard impregnating apparatus is shown in Figure 6. Impregnating treatment by vacuum-dip-pressure method can be conducted with this apparatus.

Since we can support impregnation methods and designing of impregnating apparatus such as four kinds of impregnation methods besides vacuum-dip-pressure method and the layout change of the apparatus fitted for your space, which will meet your requests, please don't hesitate to consult our sales representatives.

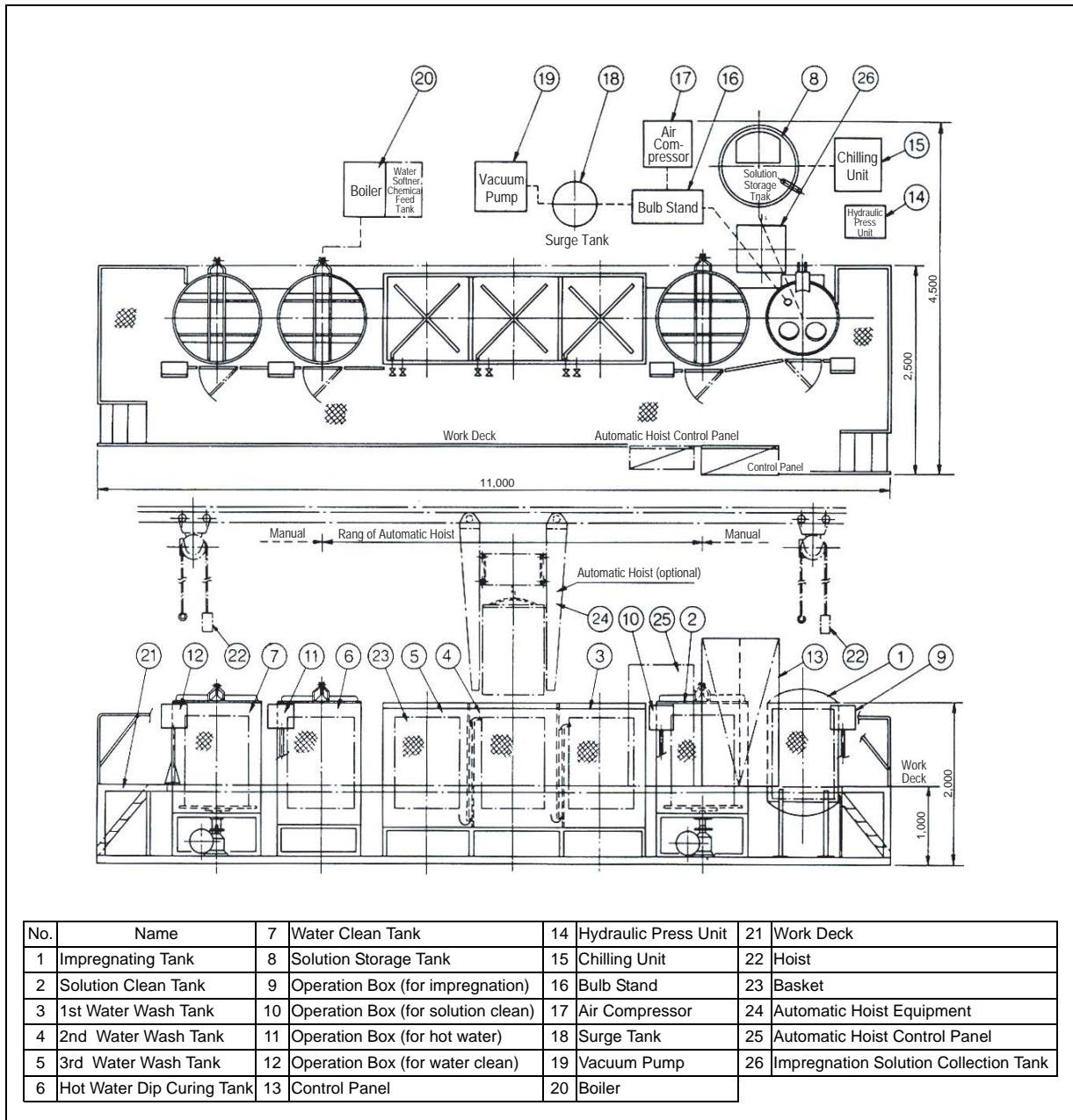


Figure 6. Impregnating apparatus

Table 4. Impregnation operation

1	Acceptance inspection	Check the state of adhesion such as oil, water, cutting trash, and stains. If they exist, conduct such as degreasing to keep it at clean state.
2	Put into basket	Put into the basket, considering the direction for drainage after impregnation.
3	Vacuum	Put the basket into the impregnation tank and retain 5 mm Hg or less of vacuum for about 10 minutes.
4	Pressurization	Infuse impregnating solution into the impregnation tank at vacuum state. Then, feed the compressed air into the impregnation tank and keep the pressure of 5 to 10/cm ² for 5 to 30 minutes.
5	Drainage	When pressure impregnation completes, return the impregnating solution in the impregnation tank to the liquid-storage tank by the pressure of compressed air. Moreover, open the lid of the impregnation tank to recover as much as impregnating solution as possible, which is adhered on the surface or dips of the object impregnated, by slanting or rotating the basket.
6	Pre-rinse	Pre-rinse with water in the rinsing tank to wash large part of impregnating solution adhered on the surface.
7	Main rinse	Wash impregnating solution down with running water and hot water.
8	Heat curing	Put in the hot air dryer for heat curing.(90°C to 130°C, 120 to 45 min.) Or, hot cure in hot water dip curing tank.(85°C to 95°C, 15 to 10 min.)

7. Application Examples of Organic Impregnation

Part of the examples of organic impregnation for leak prevention, improvement in strength, prevention of swelling after coating and plating process, and adhesive fixing are as follows:

- 1) Transport machines related examples
 - a) Cylinder block and cylinder head
 - b) Intake manifold
 - c) Torque converter case, transmission case, and crank case
 - d) Carburetor, fuel injection pump, and fuel pump
 - e) Case, bulb, and pipe for power steering system
- 2) Hydraulic equipment related examples
 - a) Body of water pump and oil pump
 - b) Body of air bulb and magnet bulb, and body of bulbs
 - c) Body of hydraulic pump and hydraulic cylinder
 - d) Cap of fire extinguisher
- 3) Pneumatic equipment related examples
 - a) Body and cover of air conditioner compressor
 - b) Body of pneumatic compressor
 - c) Air gun and spray gun
 - d) Regulator of gas, body, case, and cover of gas meter, and components of gas appliance
- 4) Others
 - a) Magnet core of sintered iron compact, sintered gear, and sintered component of iron and stainless steel
 - b) Base of machine tool
 - c) Adhesive fixing of rotor part of motor

8. Wastewater Treatment

We have described that wastewater treatment facility is needed for organic impregnation in Table 1. In the previous section of "5. Organic Impregnation Process," impregnating sealant adhered on the surface is washed out with water at "pre-rinse". Since this wastewater after rinse contains impregnating sealant, there are cases where it exceeds the standard of COD (Chemical Oxygen Demand) and BOD (biochemical oxygen demand) of wastewater. (Since wastewater standards are determined by autonomy of each region, they are not uniform across the country.)

The COD and BOD of ThreeBond solutions are shown in Figure. 7.

As shown, since the wastewater after rinse has high COD and BOD, it cannot be wasted to rivers as it is.

Therefore, wastewater treatment facility is needed to be put place side by side. Since we provide the following designing and manufacturing, please don't hesitate to consult our sales representatives:

① Wastewater incinerator, that incinerates impregnating sealants by spraying wastewater in the combustion chamber (patent is under application), ② Coagulative separation method (patent is under application), that coagulates impregnating sealant by infusing chemicals into wastewater.

Conclusion

We have summarized the organic impregnation system. Since we not only manufacture and sell impregnating sealants and impregnating apparatus but also provide processing of organic impregnation with vacuum-dip-pressure method to your products, please don't hesitate to contact our sales representatives.

At last, we add our future challenges of organic impregnation as follows:

1. The development of impregnation system from batch method to continuous method
2. The partial impregnation system, that impregnates only the places, where porosities have been generated
3. The development of wastewater facility with closed system, that does not release wastewater to the outside
4. The development of impregnating sealant, with which wastewater treatment is easy

We believe that if these are developed soon, the entire system will become compact. Therefore, easier and faster organic impregnation will become available and the demand and applications will expand further.

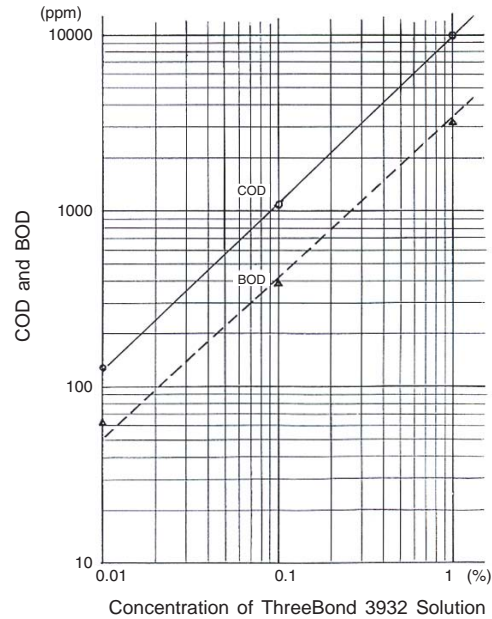


Figure 7. COD (Chemical Oxygen Demand) and BOD (Biochemical Oxygen Demand) of ThreeBond 3932 solution

« References »

- (1) International Cast Metal Defect Classification Atlas (Japan Foundrymens Society.)
- (2) Tatsuya Imoto, Surface contamination & cleaning design, 26, 10 (1985)
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- (4) R. Mc WILLIAM, 11th International Die Casting Congress and Exposition. Paper No.G-T81-075 (1981)

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