

ガス吹きプラグの開発と今後の展望

Development of Gas Purging Plug and Future Prospects

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1 緒言

ガス吹きプラグ用の耐火物は多孔質のため受鋼後、気孔に溶鋼が浸潤して気孔を閉塞し通気を阻害する問題がある。そのため酸素洗浄にてメタル浸潤層を除去するが、浸潤深さと酸洗作業がプラグを損傷させる。よって損傷の抑制、通気特性の向上を目的に開発を行ってきた。本稿では75周年以降の25年を振り返り、2000年に合併した旧ハリマセラミック(株)や2004年にアライアンス締結の旧東芝セラミックス(株)との技術集約も含めたトピックスについて、材料、構造、評価の3部構成にて記述する。

2 ガス吹きプラグの開発

2・1 材料編

26年前(1994年)当時、旧黒崎窯業(株)のプラグ用通気性耐火物は以下の3種材料系を有していた。①トップ1mmのアルミナ骨材を主原料としたシリカボンドやリン酸アルミボンドの $\text{Al}_2\text{O}_3\text{-SiO}_2\text{-Cr}_2\text{O}_3$ 系材質、②耐メタル浸潤性や耐スポール性の向上目的にトップ0.5mmのアルミナジルコニア(AZ)骨材やジルコニアムライト(ZRM)骨材を適用して、気孔径の小径化、耐食性の向上も図った $\text{Al}_2\text{O}_3\text{-SiO}_2\text{-ZrO}_2\text{-Cr}_2\text{O}_3$ 系材質、③高耐用目的でトップ1mmのアルミナ球状骨材を多用したB17材質。これは低シリカ質、高強度、耐酸素洗浄性の材質であり、ZRM骨材を高温焼成で熱分解させボンド形成材として使用している。このため従来材質に比べボンドが太く強度が高い。さらには成形かさの調整で通気と性能のバランスが取れ、顧客の多様なガス吹き条件に対応可能な材質でもある。この材質である程度の成果は得られ普通鋼メーカーを中心に、現在でも当社の主力材質の一つである。しかし特殊鋼処理では、高い操業温度

1 Introduction

Since the gas purging plugs are porous material, molten steel may infiltrate into the pores after tapping, resulting in clogging the pores and blocking gas permeation. Oxygen cleaning is applied to remove the infiltrated metal. However, when the metal infiltrated to deep inside, the oxygen cleaning gives serious damage to the plugs. We have developed technologies to reduce damaging above described manner and improve the gas permeability of the plug. In this article, we describe the development of gas purging plug during recent 25 years after 75th anniversary in the frame work of the three-part composition of materials, structure, and evaluation, including topics on technology aggregation to the merger with the former Harima Ceramic in 2000 and the alliance execution with the former Toshiba Ceramics in 2004.

2 Development of gas purging plug

2・1 Materials for the plug

26 years ago (1994), the former Krosaki Refractories possessed the following three material systems on gas-permeable refractories for the plugs. ① $\text{Al}_2\text{O}_3\text{-SiO}_2\text{-Cr}_2\text{O}_3$ system material composed by alumina aggregate with top size of 1mm as main ingredient and silica or aluminum phosphate bonds. ② $\text{Al}_2\text{O}_3\text{-SiO}_2\text{-ZrO}_2\text{-Cr}_2\text{O}_3$ system material composed by alumina zirconia (AZ) or zirconia mullite (ZRM) aggregates with top size of 0.5 mm to improve metal infiltration, spall and corrosion resistances with decreasing the pore size. ③ B17 materials composed by large amount of alumina spherical aggregates with top size of 1mm to increase the durability. This is a low silica, high-strength, and oxygen cleaning-resistant material, and the ZRM aggregates are used as a bond formation material by thermal decomposition. For this reason, the bonds are thicker and stronger than conventional materials. Additionally, by adjusting the green bulk density, balance between gas-permeability and performance can be maintained, this material can respond to the various gas blowing conditions required by the customers, with achieving a certain degree of success mainly in common steel makers, and is currently one of the main materials in our company. However, in case of special steel treatment,

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で通気性耐火物の組織変化、つまり 焼結→組織の緻密化→通気不良が発生したため耐熱性の向上が必要であった。

この時期 旧ハリマセラミック(株)と合併。2社の技術融合により開発したのが B30 材質である¹⁾。

高温焼成による組織の安定化を念頭にフラックス成分を抑制の上、メタル浸潤を極限まで抑制する目的でトップ 0.5 mm の骨材のみを使用して気孔径を小径にて均一化した。またムライト骨材適用により耐スポール特性も付与した。この材質では普通鋼処理には高位安定使用されたが、当初目的の特殊鋼処理では操業条件によってはメタルが連続して深く浸潤し通気不良となるケースも認められ、さらなる改善が求められた。

こうした背景から骨材トップを 1 mm に戻し、気孔径の大小が混在する組織で気孔径の不均一化を図るとともに耐熱性のボンド形成材の適用を検討、加えて、高温での大流量／長時間処理にも対応すべく以下のような材料設計を行った。

- ①骨材のサイズギャップによる気孔径ブロードな組織により浸潤メタルを点在化（一部通気孔の確保）
- ②ボンド部のさらなる高融点化（組織の安定化）
- ③ボンド形成材の添加量を調整し、熱間強度の適正化（メタル浸潤層の剥離～通気回復）

さらには成形性や素地強度、焼結性の向上目的に添加していた微粉を必要最小限まで削減することで特殊鋼処理時の組織の安定性を狙った。こうした点を具現化するため超高温炉を新設して B65 材質を開発した。これが実炉でも効果が認められ現在の材料開発のベースとなっている。各材質の品質一覧を表 1 に、気孔径分布を図 1 に示す。

以上の材質系と全く異なるのが低強度高通気の C01 材質である。この材質は顧客の設備や操業対応で旧東芝セラミックス(株)が製造していた材質をベースに開発したものでアルミナ破碎骨材主体の粘土ボンドの材質である。アライアンス締結後はこの材質に統一した。

as structural changes occur in porous material under high operating temperatures, in short, sintering → structural densification → reduced permeability, improvements in thermal stability were required.

At that time, the former Krosaki Refractories and the former Harima Ceramic merged. Then the B30 material¹⁾ was developed through the fusion of the technologies of both companies. With structural stability at high temperature firing in mind, and with the aim of inhibiting metal infiltration to the limit after controlling flux components, we used only aggregates with top size of 0.5 mm to homogenize the pore diameter. Additionally, by applying mullite aggregates spall resistance was improved. With this material, high-level stability was achieved for common steel treatment, but in terms of the original objective of special steel treatment, depending on the operating conditions, metal infiltrate continuously to deep inside. As a result, deterioration occurred in the permeability, so further improvements were required.

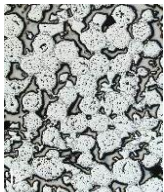
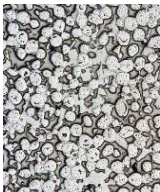
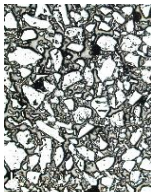

Against this background, we returned the aggregate top size to 1mm, making the pore diameter heterogenous in structures with mixture of various sizes of pore, considered to apply heat-resistant bond materials, and designed the following material to respond to high flow rates and long-term processing at high temperatures.

- ①Interspersing the metal infiltrated pore in the plug with a broad pore diameter by making the aggregate size gap (securement of permeable pore).
- ②Increasing the melting temperature of the bond (increase in the strength)
- ③Adjustment of the additives to the bond formation materials, for optimization of hot strength (peeling of metal infiltration layer to recover permeability characteristic).

Further, by reducing to the minimum of the fine powder to improve moldability, green material strength, and sinterability, we aimed to increase stability at higher temperatures for processing the special steel. To realize these points, we have newly installed a super high temperature furnace, and developed the B65 materials. The effects of this have been realized in an actual operation, and this is the base for the current material development. **Table 1** shows the property and microstructure of porous materials and **Fig. 1** shows the pore size distribution for them.

Meanwhile, the C01 material with low-strength and high-permeability is completely different from the above-described materials. This material was developed based on that originally manufactured by the former Toshiba Ceramics to support customer facilities and operations, and is clay bonded material mainly composed of the alumina crushed aggregate. After concluding the alliance, the material of this system was unified to the C01.

Table 1 Property and microstructure of porous materials

		B17	B30	C01	B65
Chemical composition / mass%	Al ₂ O ₃	91	86	91	87
	SiO ₂	3	13	6	6
	ZrO ₂	3	-	-	2
	Cr ₂ O ₃	2	-	2	4
Bulk density / g·cm ⁻³		2.94	2.65	2.49	2.6
Apparent porosity / %		23.5	30.2	33.8	31
Modulus of rupture / MPa	at R.T.	80	78	8	87
	at 1480°C	9	10	1	10
Mean pore size / μm		151	102	48	161
Microstructure					

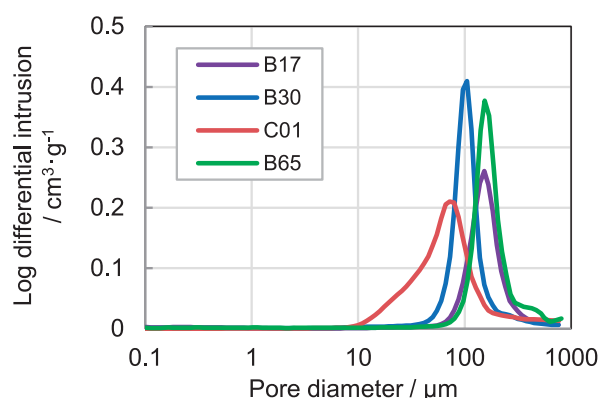


Fig. 1 Pore size distribution.

2・2 構造編

約 26 年前 (1994 年) にスリットタイプのプラグを開発した。開発にあたっては、①スリット形成材の探索、②専用のキャストブル材料開発、③製造プロセス、の 3 点を併行して実施した。①については低温で熱分解～残渣のないプラスチック材料を見出し、通気バイパスの確保に努めた。②については具備特性として 1) 耐スポール特性、2) 高熱間強度、3) 耐 FeO 性等を念頭に Al₂O₃-MgO 系の AM18SLT 材質を開発した²⁾。③では専用の鑄込み治具を作製し上下にスリット形成材であるプラスチックシートをセットした状態で鑄込みを実施した。

その後、顧客ニーズに対応すべく骨材の周囲にマイクロクラックを導入して耐スポール特性をアップした材質 (AM20SLT) や操業中のスリット形態変化をキャストブル熱膨張特性の調整により制御した AM25SLT

2・2 Structures for the plug

26 years ago (1994), the slit-type plug was developed. Upon development, following three points were implemented in parallel; ①search for materials suitable to slit forming, ②development of specialized castable materials, and ③optimizing of the manufacturing process. With regard to ①, we found out plastic materials without residue after thermal decomposition at low temperatures, and tried to secure permeable bypass. We developed Al₂O₃-MgO-type AM18SLT materials²⁾ for ②, keeping in mind to have feature of 1)anti-spalling, 2)high hot strength and 3)high resistibility against FeO. For ③, we manufactured the castings in a state where the plastic materials with slit-shaped were set at top and bottom of the plug by using a specialized casting jig.

Afterwards, to respond to customer needs, we developed AM20SLT, introducing microcracks around the aggregate to improve anti-spall characteristics, and AM25SLT, controlling slit shape changes during operation by adjusting thermal expansion characteristics of the castables. Additionally, we developed the ASP34SLTK material with both anti-spall and anti-corrosion characteristics. This material is characterized by: ①Proper strength at the intermediate temperature region with avoiding anti-corrosion degradation due to formation of low melt compounds by optimizing amount of cement added. ②Optimization of added spinel amount. ③Addition of minute amounts of organic fibers (providing anti-spalling characteristics through crack suppression). The quality list of them is shown in **Table 2**.

We also developed the segment-type plug according to the needs in overseas markets. It consists of bonded several fired bricks of thin sheet shaped with grooves like a rail on one side. Gas permeates through spaces and grooves between the sheets to molten steel. When warping or deformation occurred by firing, since the gaps become

Table 2 Composition and property of slit plug materials

	AM18 SLT	AM20 SLT	AM25 SLT	APS34 SLTK	Segment plate
Chemical composition Al_2O_3 / mass%	94	84	98.5	97.3	90.2
MgO	2.8	4	-	0.9	0.2
SiO_2	-	8.8	-	-	9.5
CaO	2	1.1	0.5	0.8	-
Bulk density / $\text{g}\cdot\text{cm}^{-3}$	3.22	2.73	3.26	3.26	2.91
Apparent porosity / %	15.3	23.4	16.5	11.3	17.7
Modulus of rupture at R.T.	150	63	125	43	87
/ MPa at 1480°C	12	1	7	14	9

等を加えた。さらに耐スポール特性と耐食性を兼ね備えた APS34SLTK 材質も開発した。これらの材質の特徴は、①セメント量の適正化による中間温度領域での強度付与、低融物生成による耐食性劣化の抑制、②添加スピネル量の適正化、③有機繊維の微量添加（亀裂抑制による耐スポール性の付与）である。品質一覧を表 2 に示す。

さらには海外市場ニーズを受けセグメントタイプのプラグも開発した。これは薄板形状の焼成れんが片面にレール状の溝を入れ数枚貼合せ、この溝から通気するプラグである。焼成による反りや変形があると溝以外の隙間が生じ各板の密着性、面接触性に劣るため厳密なる焼成管理技術を駆使して製造する。品質を表 2 に示すが Al_2O_3 - SiO_2 系の高温焼成れんがである。耐スポール性向上のため骨材部にムライトを一部適用した。さらに、通気口となる溝が 4～5 ケ所と限られているため通気不良の危険分散のため片面に 2 本ずつ溝を入れ溝の数を 8～10 ケ所と増やすことで通気特性維持の設計を実施した（図 2）。

一方でタンディッシュにおけるガス吹き上ノズル開発も行った³⁾。ここでの Ar ガス吹きは、ノズル閉塞の防止、モールド内の湯面活性化や介在物浮上促進～除去等を目的に実施される。そのためガス気泡がモールド内で浮上できず凝固シェルに捕捉されるとブローホール等の鑄片欠陥が生じる。さらに多連鑄、長時間鑄造という厳しい条件での使用が各所で定着するなか、上ノズルには更なる改善が求められている。こうした要求に応えるべく材質、構造の両面から改善を行い、実炉に適用し目的達成、鑄片品質の向上が図れている（図 3、表 3）。

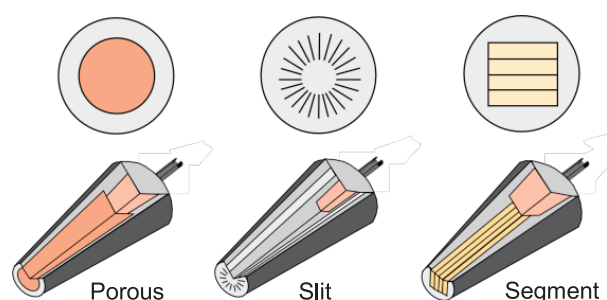


Fig. 2 Various types of gas purging plugs.

larger than the groove in the sheet, close contact of each sheet may cease. In order to avoid arising the above mentioned problems, firing process is controlled strictly. The quality is also shown in the Table 2 and this is a Al_2O_3 - SiO_2 -type high-temperature fired bricks. To improve anti-spalling property, mullite has been partly applied to the aggregate. As the number of grooves in the sheet are limited to 4-5, to diversify the risk of insufficient permeability, the groove was increased two in each sheet, resulting in increase the number to 8-10 grooves by bonding the sheets with maintaining high permeability (Fig. 2).

Meanwhile, we also developed a gas purging upper nozzle for tundish³⁾. Here, the Ar gas bubbling was implemented to prevent nozzle blockage, activate the molten steel surface in the mold, and remove the inclusions by floating action. For this reason, if the gas bubbles are unable to float and are captured in the solidifying shell in the mold, then the bubbles will cause slab defects, such as blowholes. While more severer conditions, such as multiple casting and long-term casting operations are established in various works, further improvements are required for the upper nozzle. In effort to respond to this, we have improved to reduce nozzle blockage and inclusions from both aspects of material and structure by applying to the actual operation (Table 3, Fig. 3).

Table 3 Comparison of properties in both conventional and improved TD upper nozzle materials

	Conventional		Improved	
	Porous	Dense	Porous	Dense
Chemical composition Al_2O_3 / mass%	82	86	82	87
SiO_2	7	13	7	12
ZrO_2	9	-	9	-
Cr_2O_3	1	-	1	-
Bulk density / $\text{g}\cdot\text{cm}^{-3}$	2.98	2.81	2.7	2.92
Apparent porosity / %	22.3	18.8	28	14.5
Modulus of rupture / MPa	75	105	30	130
Mean pore size / μm	23	9.5	72	3.2

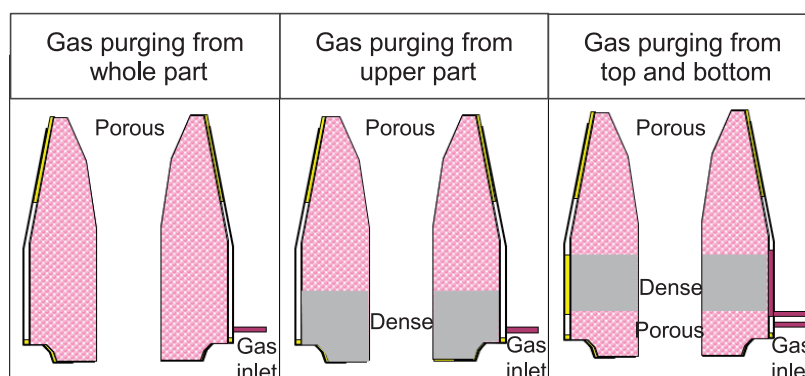


Fig. 3 Multi-layered structure of 3-type of TD upper nozzles³⁾.

2・3 評価編

評価については気孔径分布，メタル浸潤性，酸素洗浄性，X線CTによる組織観察について述べる。

2・3・1 気孔径分布

従来より水銀圧入法（JIS-R1655）にて気孔径分布を評価していたが当初は測定可能な最大気孔直径が約 150 μm 程度であり通気性耐火物の全気孔径についてカバー出来なかった。そのため，たとえ気孔率が同じ材質であっても気孔径の分布が異なるとメタル浸潤や通気特性に差が発生。よって全気孔径が測定可能な水銀圧入法装置とバブルポイント法（ASTM F316-86, JIS-K3832）装置を導入した（表 4, 図 4）。後者は液体に浸漬した通気性耐火物にガスを流し毛細管内の液体を，その細孔から押し出す圧力（この時，バブル発生）測定により貫通孔のネック部を測定するもので，両装置を併用することで，より正確な気孔径分布測定が可能となった。

2・3 Evaluation of the plug

As for the evaluation of the plug, we shall discuss on pore diameter distribution, metal infiltration, oxygen cleanability, and micro-structural observation by X-ray CT.

2・3・1 Pore diameter distribution

Previously, we had examined the pore diameter distribution using the mercury porosimetry (JIS-R1655), but the maximum pore diameter that could be measured by the method was approximately 150 μm . Namely, the method was not able to cover all the pore diameters for the porous sample. For this reason, if, for example, the pore diameter distribution is different even for materials with the same apparent porosity, differences will emerge in both characteristics on metal infiltration and permeability. Therefore, we have introduced mercury porosimetry and bubble point method (ASTM F316-86, JIS-K3832) that could cover all the pore diameters range (Table 4, Fig. 4).

The latter permeates gas into porous sample infiltrated with liquid, and by measuring the pressure (bubbles generated at that time) at which liquid within the capillaries is expelled from the pores, it is possible to measure the through hole neck section. By using both methods, it was possible to measure the pore diameter distribution much accurately.

Table 4 Comparison of evaluation methods for porosity measurement⁴⁾

	Mercury intrusion technique	Bubble point method
Target	Except for independent pore 1, pores 2, 3, 4, 5 in Fig.4	Through hole neck section Pores 3, 4, 5 in Fig.4
Principle	Mercury penetrated amount is equivalent to the volume of the pores, and the pore diameter is revealed by the mercury penetrated amount with pressure.	Gas is poured into a sample infiltrated with liquid, and by measuring the pressure that the liquid within the capillaries is expelled from the pores (bubbles are generated), the pore diameter can be measured.
Feature	General measurement method with volumetrical analyzation, suitable for the analysis of samples characterized by functioning with pore volume and porosity.	Suited to analyze the samples to pass of liquid and gas through pore . Not used mercury.

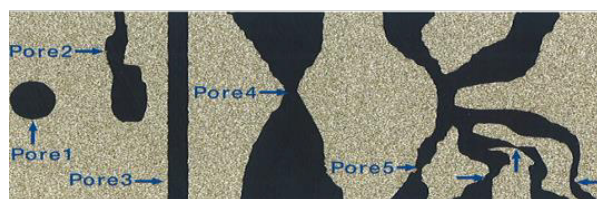


Fig. 4 Conceptual image of pore shape⁴⁾.

2・3・2 メタル浸潤性

当初は旧黒崎窯業（株）、旧ハリマセラミック（株）ともに通気性耐火物を高周波誘導加熱炉の炉底にセットして鋼種や溶鋼温度、滞留時間を変更しながらメタル浸潤深さの優劣を評価していた。しかし本方式（図 5（A））では実炉でのヘッド高さと差があり溶鋼静圧が得られず実炉結果との相関、再現には限界があった。このため九州工大 VBL と共同研究を開始して X 線透過装置を装着した電気炉（図 5（B））を用い実験温度に到達後、熔融金属に対し通気性耐火物サンプルを加圧することで溶鋼を強制浸透、その様子を録画された映像から浸潤挙動の観察及び最高浸潤深さの測定解析を実施した⁵⁾。ここで得られた知見や課題を元に、当社オリジナルな評価方法を考案、概略図を図 5（C）に示す。高周波誘導加熱炉で溶融させたメタルに、側面をシールした通気性耐火物を上部から真空引きにしながら浸漬させた（1.5 m の溶鋼静圧に相当）。浸漬深さは統一し材料ごとの相対的な評価を実施している。

2・3・2 Metal infiltration

Originally, for both the former Krosaki Refractories and Harima Ceramic, the permeable infiltration depth was measured with the conditions of changing the steel type, molten steel temperature and residence time. However, with the method shown in Fig. 5 (A), there were limits to correlate and duplicate with the actual furnace operation, since ferrostatic pressure could not be achieved due to differences in the head height with the actual furnace. For this reason, we had then started joint research with Kyushu Institute of Technology VBL, and used an electrical furnace attached with the X-ray transmission apparatus as shown in Fig. 5 (B). After reaching test temperature, by applying pressure to the molten steel, we forcibly infiltrated the molten steel into the porous sample, and observed infiltration behavior and measured and analyzed the maximum infiltration depth using the video on which this was recorded⁵⁾. Based on the knowledges and subjects obtained so far, we devised an original method of evaluation. A schematic illustration of the apparatus is shown in Fig. 5 (C). We infiltrated the molten steel in a high frequency induction furnace while evacuating the porous sample sealed with the side (equivalent to the ferrostatic pressure of 1.5 m depth). We performed a relative evaluation for each material, with making the infiltration depth constant.

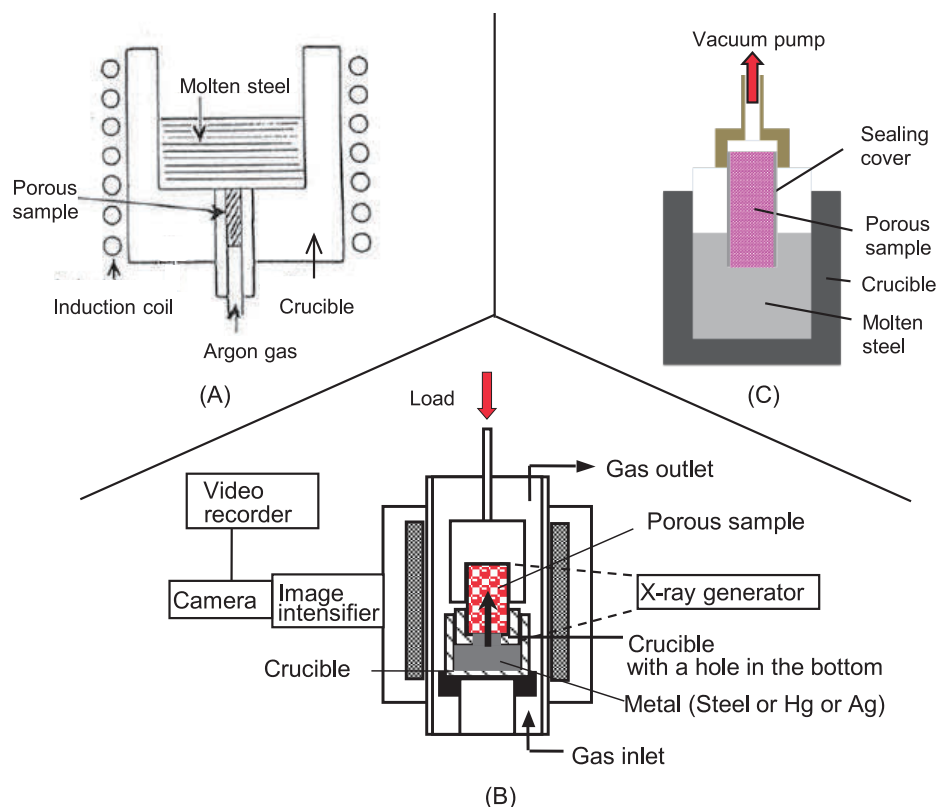


Fig. 5 Schematic illustrations of molten steel penetration tests (A),(B) and (C)^{2,5,6}.

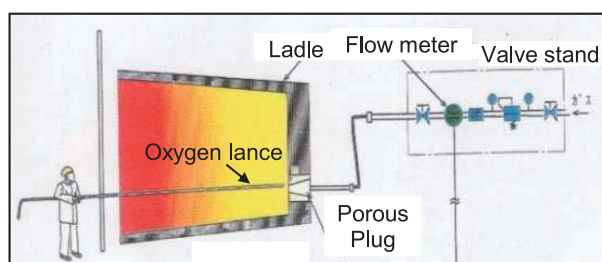


Fig. 6 Appearance of oxygen cleaning in actual operation.

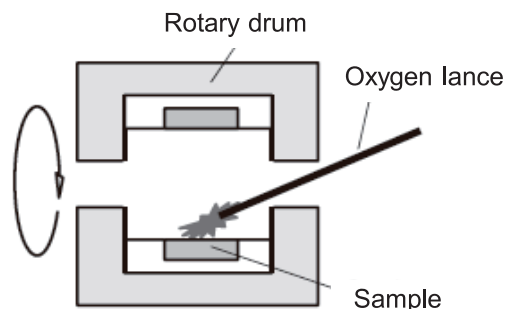


Fig. 7 Schematic illustration of the cross section of oxygen cleaning test.

2・3・3 酸素洗浄性

回転浸食炉内に通気性耐火物を内張りして所定の温度到達後、実炉同様ランスによる酸素ガス吹き込み（図 6）により損傷状態を評価している。これは旧ハリマセラミック（株）方式であり、これで材料ごとに耐酸素洗浄性を相対的に比較評価している（図 7）。

2・3・4 X線CTによる組織観察

通常の組織観察、EPMA 分析という二次元解析に加え、X線CTにより通気性耐火物を三次元的に解

2・3・3 Oxygen cleanability

In actual use, permeability of gas purging plug was maintained by cleaning with oxygen gas blow (Fig. 6). Therefore, after the designated temperature reached, we evaluated the damage caused by an oxygen gas blow from lances like actual use after lining the inside of a rotary erosion furnace with permeable refractories. This is the former Harima Ceramic method and we used this to test a comparative performance of the oxygen cleanability resistance per material (Fig. 7).

2・3・4 Micro-structural observation using X-ray CT (computerized tomography)

In addition to the ordinary micro-structural observation, and two-dimensional analysis known as

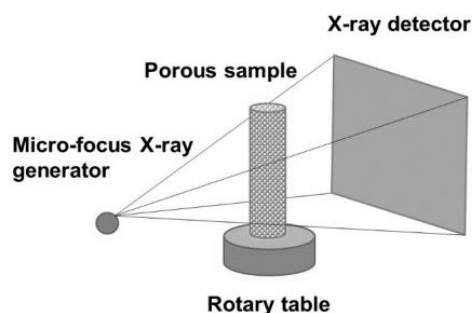


Fig. 8 Schematic image of 3-dimensional X-ray CT⁶⁾.

析し気孔構造の可視化を可能としている⁶⁾。目的は、気孔構造をより詳細明確に把握することが耐金属浸潤性に繋がり、酸素洗浄による損傷も極力抑制できるためである。図8にX線CTシステムの概念図を示す。断層画像はターゲットから放出されたX線が回転ステージ上のポーラスサンプルを透過し、後方に置かれたX線検出器に二次元の投影像を投射することで取得。得られた断層画像を元に気孔部分を抽出して三次元構成を行うことで気孔構造の可視化を実施した⁶⁾。

図9は実際のポーラスサンプルにおける気孔の三次元構造である。白い部分が気孔を示しており、骨材と気孔を区別してサンプル内を可視化できる。こうした中、2・3・2 金属浸潤性、で評価済みのサンプルについても画像解析を行い金属の浸潤状況を調査することで耐金属浸潤性に効果がある気孔構造を見出し開発に繋げている⁶⁾。

3 今後の展望

この25年を振り返ると、合併やアライアンスによる技術の相乗効果に加え高温焼成炉、各種測定評価装置の導入、充実などソフトとハードの両面で格段の進歩があった。今後は実炉結果を元にガス吹きプラグのあるべき姿を念頭に、実炉と関連ある金属浸潤性や酸素洗浄性、通気回復性の確認とX線CTによる組織の定量的解析を有機的に進め、理想とする多孔組織を見極め実形状製造に反映させ、常に高位安定で顧客満足が持続可能な究極のガス吹きプラグを目指していく。

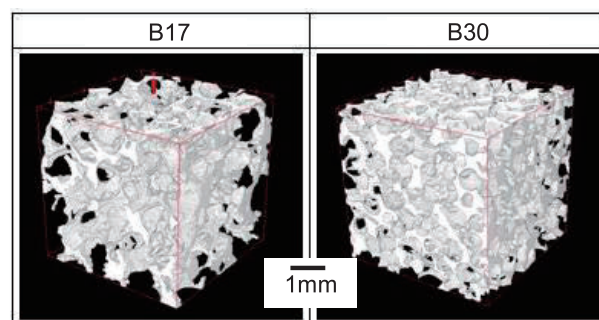


Fig. 9 3D structure of pores in the porous sample⁶⁾.

EPMA, 3D analysis of the porous sample using X-ray CT made possible to visualize the porous structure⁶⁾. The goal is that by grasping the detail of the porous structure in more accurately, this will link to improvement of the metal infiltration resistance, and the damage caused by oxygen cleaning can be minimized. A schematic diagram of the X-ray CT system is shown in Fig. 8. The tomographic image is obtained by the X-ray discharged from the target passing through the porous sample at the rotation stage, and the 2D image being projected on to an X-ray detector placed at the back⁶⁾.

Figure 9 shows the 3D structure of pores in the porous sample. The white section shows the pores, and the aggregate and pores can be differentiated and visualized within the sample. Under such, an image analysis was performed on the samples on which metal infiltration was discussed in 2・3・2, and by surveying the metal infiltration status, we could discover the porous structure that was effective to increase the metal infiltration resistance, and this linked to our development⁶⁾.

3 Future Outlook

When looking back over these 25 years, gas purging plugs has significantly advanced both in soft and hard aspects with installation and enhancement of high temperature firing furnace and various apparatus for testing. In the future, keep in mind the ideal commercial goods as gas purging plug based on the effects of actual use, we will aim to organically progress in confirming metal infiltration, oxygen cleanability and permeability recovery by using a method with correlative evaluation on actual use, performing quantitative structural analysis using the X-ray CT. We will intend to develop the extreme gas purging plug that will consistently satisfy our customers with its high level of stability.

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