Characterization of the Open Porosity of Brake Pads. III. Analysis of the Effects of a Thermal Treatment on Porosity.

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ABSTRACT

Mercury porosimetry, 2-D image analysis and 3-D surface microanalysis techniques were used to study changes in the porosity of brake pads after a thermal surface treatment. Mercury porosimetry showed that the porosity increased with the intensity of the thermal treatment. This increase was especially large for pores with a diameter of more than 10 μ m. 3-D microanalysis revealed holes on the treated surface which became increasingly open and deep. Image analysis was used to establish the porosity distribution according to depth, and revealed that the thermal treatment caused changes in the porosity to a limited depth in the pad. The extent of this zone depended on the intensity of the treatment, but was not related to the pad's initial porosity.

1. INTRODUCTION

In Parts I /1/ and II /2/, we described the methods used to characterize the open porosity of brake pads. Pore surface area parameters, measured using an image analysis technique on oriented sections, were correlated with the porosities measured by mercury porosimetry to obtain correlations characteristic of the open porosity of the friction materials studied. This third part is devoted

to applying these methods to the study of the changes in porosity after the pad surfaces are subjected to a thermal pre-grinding treatment. Effective pre-grinding should simulate the state of the pad in the area surrounding the friction surface after that surface has been subjected to the high temperatures produced by braking.

2. THE THREE BASIC BRAKE PAD TYPES

The studies were conducted using 3 types of pads which were produced from the same basic A0 formula (a single initial blend of constituents, the composition of which will not be disclosed here) and which are characterized in Parts I /1/ and II /2/.. These 3 types of pads are called A0P10, A0P15 and A0P20, since their respective target porosities were 10%, 15% and 20%. Actual porosities were obtained by applying various amounts of pressure during production with all other parameters identical. Standardized thermal treatments of increasing intensity (V2, V3 and V5) were used on the three types of A0P pad and so, for the A0P10 pad, for example, they will be referred to as A0P10 V2, A0P10 V3, A0P10 V5 and A0P10 NT for the pad not treated.

3. STUDY OF THE EFFECTS OF THE THERMAL TREATMENT ON POROSITY USING MERCURY POROSIMETRY

Table III-1 lists the average values of the porosities measured by mercury porosimetry. The thermal surface treatment produced changes in the overall porosity, which increased with the degree of treatment from Not Treated (NT) to intensity V5.

The mercury porosimetry analysis of the untreated pads revealed three porosity ranges: range 1 (low volume, and made up of several wide openings near the surface); range 2 (intergranular porosity); range 3 (the intragranular porosity of certain constituents) /1/.

As an example, as the chart for A0P10 pad shows below (Figure III-1), the open porosity increased as a function of the growing intensity of the thermal surface

Table III-1
Porosity volume percentages for pads A0P10, A0P15 and A0P20, untreated (NT) and with V2, V3 and V5 treatments.

	NT	V 2	V3	V 5
A0P10	12.0	13.1	13.7	15.4
A0P15	15.8	15.9	16.2	16.7
A0P20	19.8	20.5	20.8	22.7

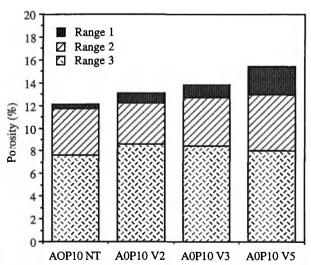


Fig. III-1: Porosity distribution for A0P10, untreated (NT) and with treatments V2, V3, and V5.

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treatment, with a particular increase in range 1 porosity, characteristic of the openings near the surface. On the other hand, for ranges 2 and 3, no significant quantitative changes appeared, but this does not exclude compensating structural changes in the relevant porosities, as revealed by image analysis of sections perpendicular to the treatment surface.

The porosity was also measured for the first 5 millimeters of thickness of each of the treated surfaces. The relative increases (%) in porosity as a function of the thermal treatment as measured for the entire pad and for the first 5 millimeters of thickness starting from the treated surface are shown in Table III-2. It can be seen that, for each type of pad and for each thermal treatment, the increase in porosity was more significant for the first 5 millimeters than for the material as a whole. As was the case with the pads taken as a whole, the porosity increased in the first 5 millimeters as a function of the intensity of the thermal treatment. However, as was shown by the 2-D image analysis of the sections perpendicular to thermal treatment surface, these are overall results and they cannot be taken to describe the distribution from the surface inward of the changes in the initial porosity, and in particular, the thermal treatments cannot be correlated with the initial porosity. Nevertheless, it did appear that the smaller the initial open porosity, the bigger the increase in porosity turned out to be.

So the surface treatment caused changes in the porosity, particularly in the area near the surface.

4. STUDY OF THE EFFECTS OF THE THERMAL TREATMENT ON POROSITY USING 3-D SURFACE MICROANALYSIS

To quantify the changes resulting from the surface treatment, a 3-D surface microanalysis of the A0P15 NT, V2, V3 and V5 pads was conducted using a diamond-tipped feeler (radius of curvature: 2.5 μm) designed in the laboratory /3,4/. The movement of the sample in relation to the sensing instrument in the X and Y directions (surface) and of the instrument in relation to the sample in the Z direction (height) was accomplished using three electronically controlled motorized tables run by a microcomputer. The coordinates (X, Y, Z) of each point of the surface were

		V 2	V3	V 5
A0P10	10 m m	8.7	13.8	28.0
	5 m m	23.6	35.7	45.0
A0P15	10 m m	1.3	2.9	5.9
	5 m m	8.9	13.4	29.3
A0P20	10 m m	3.9	5.2	14.8
	5 m m	7.1	11.1	30.3

Table III-2

measured by scanning the sample over 256 lines of 256 points each, for a total of 65,536 points.

The four surfaces studied were analyzed with an increment of 60 µm on the X and Y axes, which makes for a total analyzed surface area of 15.3 mm x 15.3 mm, or 234.1 mm². Figure III-2 shows Williamson's reversed perspectives /5/ for the A0P15 NT, V2, V3 and V5 pad surfaces so as to bring out the holes (porosity of

the pad surface). The A0P15 NT surface showed very little roughness and few holes. The surface of A0P15 V2 was not much different from that of A0P15 NT. The surfaces of the A0P15 V3 and V5 pads were much rougher and were clearly distinguished from the A0P15 NT and V2 surfaces in that there were numerous holes.

From A0P15 NT to A0P15 V5, the peak-to-valley height increased, which was due to the elimination of

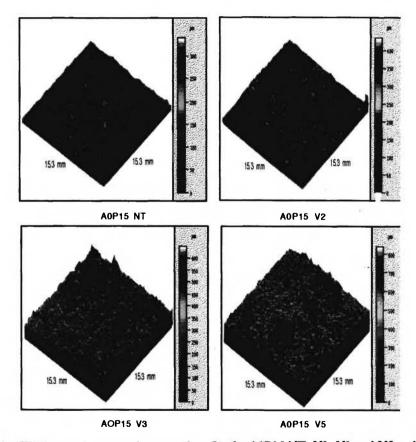


Fig. III-2: Williamson's reversed perspectives for the A0P15 NT, V2, V3 and V5 pad surfaces.

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certain constituents by decomposition over an evergreater depth, with the development of increasingly open and deep holes.

The numerical values corresponding to the proportion of the surface (points scanned) in various depth ranges are shown in Table III-3. The majority of the surface points of A0P15 NT and A0P15 V2 were at a depth of between 100 μ m and 200 μ m, while the majority of points for A0P15 V3 and A0P15 V5 were at 300-400 μ m and 400-500 μ m, respectively.

Hence, the changes in the surface roughness increased with the intensity of the thermal treatment.

5. STUDY OF THE EFFECTS OF THE THERMAL TREATMENT ON POROSITY USING IMAGE ANALYSIS

The A0P15 V5, A0P15 V3 and A0P10 V5 pads were selected for a study of the influence of the intensity of the surface treatment on the porosity distribution according to the depth, in addition to the relation between the initial porosity and the effects of the treatment.

Using a section perpendicular to the thermally treated surface, each sample was analyzed on a series of bands parallel to the surface. In each band, two parallel lines broken down into 16 measurement fields each were analyzed, thus providing porosity measurements for increments of about every 250 μ m of depth (Figure III-3).

For each pad, the distribution of the surface area porosity according to depth was determined for two sets of pores: all pores, and only those pores with an area of

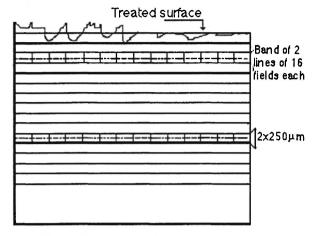


Fig. III-3: Diagram of the image analysis of the treated pads (section perpendicular to the treated surface).

less than $10,000 \mu m^2$ (the maximum pore surface measured on untreated pads).

Figure III-4 shows the surface area porosity distribution according to depth for A0P15 V5 and A0P15 V3. The surface area porosity was greatest at the level of the treated surface, then it decreased. The thermal treatment caused changes in the porosity to a greater depth for A0P15 V5 (5 mm) than for A0P15 V3 (2.5 mm). Indeed, below these values, the surface area porosity varied little and remained close to that of an untreated pad.

The zone affected by the treatment can be broken down into two ranges. In the first range (A0P15 V5: 0-3 mm; A0P15 V3: 0-0.8 mm), the falling porosity curve was due principally to the decrease in the number of pores with a surface area of more than $10,000 \, \mu m^2$.

Table III-1
Porosity volume percentages for pads A0P10, A0P15 and A0P20, untreated (NT) and with V2, V3 and V5 treatments.

A0P15	NT	V 2	V 3	V 5
Depth ranges				
(µm)				
0-100	11.5	10.6	0.03	0.01
100-200	87.7	83.3	0.15	0.01
200-300	0.8	7.0	25.3	0.04
300-400		0.9	67.8	1.1
400-500			6.2	61.2
>500			0.4	37.6

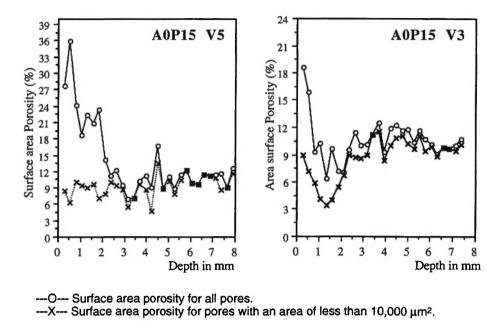


Fig. III-4: Distribution of surface area porosity according to depth for the A0P15 V5 and the A0P15 V3 pads.

In fact, for the pores with a surface of less than 10,000 μm^2 , the porosity stayed practically constant or declined very slightly. In the second range (A0P15 V5: 3 - 5 mm; A0P15 V3: 0.8 - 2.5 mm), pores with large surface areas became rather rare. The porosity of the pores with a surface area of less than 10,000 μm^2 was lower than that measured in the part of the pad not affected by the thermal treatment. This can be explained by the fact that the initial pores were partially sealed up with tars produced during the treatment. The progression was similar for pores with a surface area of less than 10,000 μm^2 in the first range, but in this case, this was due to the fact that large pores were created in part to the detriment of smaller pores.

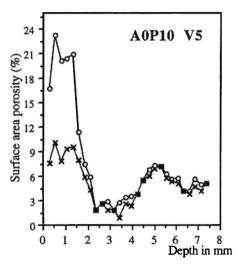
It is possible to obtain a computed volume porosity for the first 5 millimeters of the thermally treated pads by applying the correlation between the measurements obtained by mercury porosimetry and those obtained by image analysis /2/. For example, the average surface area porosity for A0P15 V5 was 15.81%, which corresponds to a volume porosity of 20.5%. This value, which was very close to that obtained by mercury porosimetry (20.3%), was thus in keeping with the correlation characteristic of the porous structure of these brake pads. This would tend to prove that although the

thermal treatment changed the dimensions of the pores, it did not fundamentally alter the "interconnected ink bottle" structure of the intergranular porosity /2/.

The distribution of the porosity of A0P10 V5 according to depth was only slightly different from that of A0P15 V5 (Figure III-5), which showed that the effects of a thermal treatment of a given intensity depended little on the initial porosity.

As the photomontage in Figure III-6 (first 6.5 mm from the treated surface) shows, the porosity distribution according to depth for A0P10 V5 can be broken down into 3 zones corresponding to those that show up in Figure III-5:

- The first 2.2 mm of depth a high-porosity zone characterized by pores with a large surface area.
- The next 2.2 mm a low-porosity zone. The large light-colored spot in the photo is a particle of rubber. In this zone, the rubber particles gave off a continuous green fluorescence because of the fluorescent resin impregnated in the microporosity of this constituent /1/. On the other hand, in the zones not affected by the thermal treatment, the rubber had a very low level of fluorescence, which made it show up as a brown color under UV



- --O--- Surface area porosity for all pores.
- ---X--- Surface area porosity for pores with an area of less than 10,000 μm².

Fig. III-5: Distribution of the surface area porosity of A0P10 V5 according to depth.

illumination. Moreover, the rubber has an internal porosity which appears in the form of large cavities that disappear during the thermal treatment. So in this low-porosity zone, the rubber underwent partial chemical decomposition, probably accompanied by degassing by the small pores as they formed.

 Finally, a zone corresponding to the area not affected by the thermal treatment.

CONCLUSION

This study made it possible to quantify the influence of a thermal surface treatment on the porosity of AOP-type brake pads. On a general level, the treatment created new porosity zones. In the first zone, there was significant decomposition of certain constituents, accompanied by the disappearance of their initial small-pore porosity and the appearance of large pores. Increasing the intensity of the thermal treatment resulted in this new porosity becoming more significant and its distribution zone becoming deeper.

In the second subjacent zone, the decomposition was less significant, and tars sealed up part of the initial porosity. Nevertheless, intergranular porosity seemed to retain the same fundamental structure (chains of interconnected "ink bottles" around rigid grains which cannot be broken down thermally), although the dimensions increased. These measurements could now be used to quantify the porosity of used brake pads with the same methods in order to validate a pre-grinding technique.

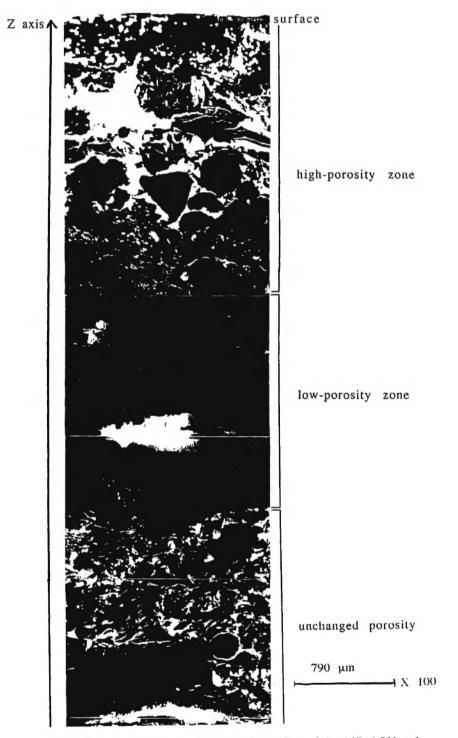


Fig. III-6: Section perpendicular to the surface of the A0P10 V5 pad.

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