Surface glazing of concrete using a 2.5 kW high power diode laser and the effects of large beam geometry

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ABSTRACT

Interaction of a 2.5 kW high power diode laser (HPDL) beam with the ordinary Portland cement (OPC) surface of concrete has been investigated, resulting in the generation of a tough, inexpensive amorphous glaze. Life assessment testing revealed that the OPC glaze had an increase in wear life of 1.3 to 14.8 times over an untreated OPC surface, depending upon the corrosive environment. Also, variations in the width of the HPDL beam were seen to have a considerable affect on the melt depth. Furthermore, the maximum coverage rate that it may be possible to achieve using the HPDL was calculated as being 1.94 m^2/h. It is a distinct possibility that the economic and material benefits to be gained from the deployment of such an effective and efficient large area coating on OPC could be significant.

Keywords: High power diode laser; Concrete; Ordinary Portland cement; Surface; Glazing

1. Introduction

Owing to their unique characteristics, lasers have the propensity to be employed for the non-contact processing of materials which are otherwise difficult to process. Concrete is a composite material consisting of an array of fine and coarse aggregate pieces embedded within an ordinary Portland cement (OPC) matrix. Consequently the processing and surface treatment of OPC surface of concrete can be an arduous task.

To date, many studies have been carried out to investigate the laser processing of concrete. Most of the research, however, has concentrated on the laser cutting of concrete and reinforced concrete using high power CO_2 lasers, most prominently with regard to nuclear reactor decommissioning [1-3]. Also, as part of nuclear plant decommissioning, Li et al. [4-7] conducted much research to determine the workability of several laser techniques for sealing/fixing radioactive contamination onto concrete surfaces. Such techniques experimented with were: direct glazing of the concrete, single and multiple layer fusion cladding and combined chemical/fusion cladding. In addition, Johnston et al. [8] have reported on the successful removal of the surface layer of concrete (scabbling) by means of Nd:YAG and CO_2 laser radiation. Work by Sugimoto et al. [9] focused upon modifying the surface appearance and surface properties of cement based materials using a high power CO_2 laser. The laser treatment produced novel surfaces, with surface textures, properties and appearance unique to laser treatment. The resultant physical characteristics and mechanical behaviour of the post-process cement based materials was later fully characterised by Wignarajah et al. [10]. Borodina et al. [11] has carried out investigations into the structural changes within the composition of zirconia concrete caused by surface exposure to CO_2 laser radiation, detailing microstructural changes, phase changes and the absorptivity characteristics. In all of these studies, spallation and excessive cracking and porosity formation were found to be major problems undermining the performance of the laser treated surface layer.

This present work is concerned with the utilisation of the relatively novel 2.5 kW high power diode laser (HPDL) to generate a surface glaze on the OPC surface layer of concrete, and the effects thereof on the OPC’s mechanical, physical and chemical properties. In addition, the unique effects of processing using a relatively large beam geometry are examined. The ultimate aim of this investigation is to facilitate the hitherto impossible task of effectively generating a durable and long-lasting surface seal on the concrete, thereby extending the life and applications base of the concrete.

2. Experimental procedure

The laser used in the study was a 2.5 kW HPDL (Rofin-Sinar, DL-025), emitting at 940nm. The defocused laser beam was fired directly onto the samples with rectangular beams of up to 6mm x 40mm with powers ranging from 0.5-2.5 kW. The laser head assembly and focusing optics are shown schematically in Fig. 1. The beam was traversed across the samples by means of mounting the assembly head onto the z-axis of a 3-axis CNC table. The defocused laser beam was thus fired across the 'as cast' OPC surface of the concrete by traversing the samples beneath the laser beam using the x- and y-axis of the CNC table at speeds of 240-1800 mm/min, whilst 20 l/min of coaxially blown Ar gas was used to shield the laser optics.

The concrete studied in the experiments was the ubiquitous OPC based concrete. For the purpose of experimental convenience the as-received concrete blocks were sectioned into squares (120 x 120 x 20mm) prior to laser treatment. The composition of the concrete by volume is as follows: 20mm limestone aggregate (40%), 10mm limestone aggregate (14 %), zone M sand (28.5%), OPC (10.5%) and particulate fine aggregate (7%).

To determine the characteristics of the glazes, the HPDL treated concrete samples were examined using optical microscopy. Mechanical and chemical testing of the OPC glaze in comparison with the untreated OPC surface of concrete was conducted to determine such properties as pull-off (bond) strength, rupture strength and wear resistance. Additionally, chemical tests were carried out to examine the corrosion resistance of the laser glazed and untreated OPC with regard to acid (nitric acid) alkali (sodium hydroxide) and common industrial detergent. Life assessment testing of the laser glazed and untreated OPC was also carried out.

3. Laser glazing operating parameters

The OPC surface of the concrete was seen to glaze readily when exposed to a wide range of HPDL power densities and traverse speeds. However, to accurately asses the propensity of the OPC surface of the concrete to laser glaze, the relationship between the power density and the interaction time was studied. The results of this investigation are given in Fig. 2.

It is evident from Fig. 2 that a power density minimum of around 1.25 kW/cm^2 exists, below which glazing will not occur, regardless of the interaction time; whilst the minimum interaction time, below which a significant increase in power is required to produce glazing, is around 0.3 seconds.

Fig. 3 schematically illustrates the concrete laser glazing operating window in terms of traverse speed and power density. Within the optimum operating conditions good quality glazes displaying few porosities and microcracks could be produced. Furthermore, from Fig. 3 it is possible to ascertain the maximum coverage rate that it may be possible to achieve using the HPDL. This was calculated as being 1.94 m^2/h for a rectangular beam of 6mm x 30mm with a laser power of 2.5 kW and a traverse speed of 720 mm/min.

4. Morphological characteristics of the laser glaze

4.1 Glaze formation mechanisms

The chemistry of the OPC surface of concrete and the hydration of its various constituents is extremely complex, and as such is not yet fully resolved [12]. Nevertheless, it is known that the constituents of OPC are minerals which exist as multi-component solid solution chemical compounds. Of particular importance with regards this study, OPC contains in relatively large proportions: SiO_2 (21wt%), Al_2O_3 (5wt%) and Fe_2O_3 (3wt%), which are basic glass network formers and modifiers. Consequently the intense local heating brought about by the incident HPDL beam results in the melting of these compounds at around 1283^{0}C, thereby causing the materials to lose the retained water and form an amorphous glassy material consisting of various calcium-silicate-alumina compounds [4]. Indeed, the amorphous nature of the HPDL generated glazes was verified by an XRD analysis.

As was mentioned earlier, HPDL interaction with the OPC surface occasioned a dramatic colour change; changing from grey to green. Such a change can be ascribed to the resultant phase transitions, and also the presence in small concentrations of metal transition ions in various oxidation states within the OPC composition, in particular, ferric ions in the Fe^{3+} and Fe^{2+} oxidation state. Fe^{3+} and Fe^{2+} ions are known to give rise to green and blue colours respectively when subjected to intense heating [13, 14]. However, if both phases are present within the composition, then the colour is determined by the Fe^{3+}/Fe^{2+} ion ratio, resulting in dark blue or black colours [13, 14]. Since the surface produced after HPDL treatment was green, then it is reasonable to assume that both phases were not present within the OPC.

4.2 Cracking and porosity formation

As Fig. 4 shows, cracking of the HPDL induced glaze occurred. The formation of cracks can be attributed mainly to thermal stresses generated during laser irradiation. This is due to the fact that OPC has low thermal conductivity, and, as such, during laser heating a large thermal gradient between the melt zone and the substrate exists which results in the generation of thermal stresses. Additionally, despite the fact that the laser surface treatment process is effectively localised in nature, the fact remains that a certain amount of the heat generated will be conducted to sections of the OPC where the surface is already glazed. This, combined with existence of a relatively cold OPC substrate means that thermal stresses will be generated. During the heating phase the stresses will be compressive and relieved by plastic deformation, thus precluding crack formation. At high temperatures ($T \geq T_m$) the stresses can also be relieved [15-17]. However, during cooling when the temperature falls below T_m, then stresses will accumulate. If the fracture strength of the material is exceeded, then cracking within the melted layer will occur. The thermal stress σ, induced by a thermal gradient can be calculated using the Kingery equation:

$$\sigma = \frac{E\alpha\Delta T}{1-\upsilon} \tag{1}$$

where E is Young's modulus, ΔT is the temperature change, α is the coefficient of thermal expansion and ν is Poisson's ratio. More succinctly, ΔT is the difference between the critical temperature (below which stresses can no longer be relieved) and ambient temperature. For OPC this is the difference between the melting point, 1283^0C and ambient temperature 20^0C. So, if it is assumed that the glass formed on the surface of the OPC is similar to soda-lime-silica glass because the compositions of the two materials are similar, then by using the following values for pyrex: E=6.42 x 10^4 MN m^{-2}, α=33 x 10^{-7} K^{-1}, ΔT=1263^0C and ν=0.176, when the OPC surface of the concrete was irradiated by the HPDL beam the thermal stress produced in the resulting glass according to Eq. (1) was around 305 MN m^{-2}. Since this is well in excess of the fracture strength of the glass, 120 MN m^{-2} [18], cracking will occur, and can only be avoided by severe distortion or through the reduction of ΔT by pre-heating.

From Fig. 4 it can be seen that porosities were a common feature of the HPDL induced glaze, varying in size from microscopic pits to large craters depending upon both the laser operating parameters and the actual laser employed. For all instances of porosity formation the mechanism behind their development is the consequence of gas escaping from within the melt and disrupting the surface [19]. With regard to the OPC glaze, the gas is likely to be CO_2 [10]. If the laser energy density incident on the OPC is too low, then the generated CO_2 can not escape from the molten OPC surface easily because of the high viscosity of the melt. As such, when the CO_2 gas eventually does penetrate the melt surface, the resultant porosity is not filled by the flow of the melt; since the insufficient energy density is unable to maintain a high enough temperature for an adequate length of time and thus decrease the overall viscosity of the melt [20]. In this case the porosities formed are typically small and shallow, being regular in both periodicity and intensity . On the other hand, if the laser energy density incident on the OPC surface of the concrete is too high, then boiling of the surface may happen. At the same time an increase in CO_2 gas formation may occur within the melt. These individual pockets of CO_2 gas formation may combine and rise to surface of the melt. Once the energy density decreases (as the laser traverses away), then the additional CO_2 gas will attempt to escape from the molten surface. However, the solidifying melt will prevent this, causing bubbles to form. The excessive CO_2 gas pressure will firstly cause the bubbles to expand and ultimately rupture the walls of the bubbles creating a sharp ‘knife edge’ porosity [3, 10]. These types of porosity are usually large, deep and randomly spaced.

5. Mechanical, chemical and physical properties

Current British and international standards in relation to concrete are concerned only with water absorption and compressive strength. Consequently, it was not possible to test the HPDL generated glazes according to, and strictly adhering to, established tests. As such, wherever possible tests based on current standards were developed to investigate specific aspects of particular relevance to the HPDL generated glazes, namely: pull-off strength, surface roughness, rupture strength, wear resistance and corrosion resistance.

5.1 Pull-off strength

To assess the strength of the bond between the HPDL generated surface glaze on the OPC and the concrete substrate itself, pull-off tests were conducted. For the tests the concrete was prepared as relatively small area samples (25mm x 25mm). High tensile aluminium test dollies were then attached onto the glazed surface and to the axially opposite concrete substrate surface using Araldite epoxy and left to cure for 24 hours. In order to ensure axial accuracy (essential for true results), the test dollies were set in position using identical V-blocks. The samples were placed into an Instron 4507 tensile/compressive test rig by mounting the test dollies into the jaws of the rig. A tensile force was then applied until failure with the energy being simultaneously recorded.

The results obtained varied markedly with changes in the laser operating parameters, as Fig. 5 shows. A post-test analysis of the samples showed that the material failed approximately below the laser treated surface in the heat affected zone (HAZ). Within the optimum laser operating parameters the average bond strength of the glaze was recorded as 24 MPa. This compares with 63 MPa for the untreated OPC surface of concrete.

5.2 Rupture strength

Tests were conducted to determine the rupture strength of the OPC glaze. Test samples were prepared as described above. The samples were placed onto the sample stage of the Instron 4507 tensile/compressive test rig and then subjected to a compressive rupture force until the OPC glaze failed (cracked), with the energy being simultaneously recorded. The rupture force was applied by means of a high tensile steel indentor with a 1mm radius point. The results of the tests revealed that the average rupture strength of the OPC glaze was only 0.8 J, whilst the rupture strength of the untreated OPC surface was some 4.3 J.

5.3 Wear life characteristics

The wear resistance of a material in general is determined primarily by the hardness of the material in comparison with the hardness of other materials with which it comes into contact [21]. However, wear resistance does not always increase with hardness [22]. Tests were therefore conducted to determine the exact difference in wear resistance characteristics of the OPC glaze and the untreated OPC surface. For experimental purposes the OPC was cut into smaller pieces (25 x 25mm). Half of the samples were then laser treated. All the samples were then weighed and subjected to a friction force for 8 hours, being removed from the machine and weighed at two hourly intervals.

Fig. 6 shows the relationship between weight loss and the friction time for the OPC glaze and the untreated OPC. As one can see, the OPC glaze shows a significant increase in wear resistance over the untreated OPC surface, with the weight loss being 2 times lower after 4 hours, and 3 times lower after 8 hours.

5.4 Corrosion resistance

Concrete surfaces are often subjected to corrosive substances, either as part of the normal service environment and/or as a result of routine cleaning. Therefore corrosion resistance tests based upon BS 6431 [23] were conducted using nitric acid, sodium hydroxide and Premier Products MP9 detergent cleaner. The experiments were carried out by dropping small amounts of the corrosive agents in the concentration ratios of 80%, 60%, 40%, 20% and 10% on to the surface of the untreated and HPDL glazed OPC surface of concrete at hourly intervals for four hours. The samples were then examined optically and mechanically tested in terms of compressive strength and wear. High concentrations of the various corrosive agents were used principally to accelerate the tests. However, in practice 60% nitric acid is used within the nuclear processing industry as a solvent for nuclear fuels [24].

All three substances in the concentrations 80%, 60% and 40% were seen to immediately attack the untreated OPC surface, with the nitric acid and sodium hydroxide attacking with greater severity than the detergent, whilst the HPDL glazed surface displayed no discernible microstructural changes or signs of devitrification due to corrosion.

Tests conducted according to ASTM C579-91 [25] revealed that exposure of the untreated OPC surface to the reagents had a significant effect on the compressive strength and the wear resistance of the OPC. Exposure of the OPC to nitric acid and sodium hydroxide in the concentrations 40-80% resulted in an average loss of compressive strength of approximately 19-37%. In the case of the detergent a discernible loss in compressive strength only occurred with concentrations above 40%. Here the average loss in compressive strength for concentrations in the range 60-80% was approximately 17%. This compares with no discernible difference in either the wear resistance or the compressive strength of the HPDL glazed surface.

Fig. 7 shows the variation in wear resistance of the untreated OPC surface when exposed to the reagents with an 80% concentration. As one can see, the wear resistance is significantly affected, particularly through interaction with the nitric acid and the sodium hydroxide. Here the weight loss was approximately 5 times higher than for the unexposed OPC after 4 hours, and approximately 11 times higher after 8 hours for the nitric acid. In the case of the detergent the weight loss was marginal after both 4 and 8 hours.

6. Effects of comparatively large laser beam geometry

As discussed previously, the use of the 2.5 kW HPDL offered for the first time the possibility glazing the OPC surface of concrete at very high rates. Such an achievement was facilitated primarily by the possibility of employing relatively large geometry beams (up to 6 x 40mm). However, as the work of Liu et al. [26] revealed, the use of relatively large laser beams is not without attendant problems, not least the generation of melt pools exhibiting deep central sections.

Experiments to investigate this disadvantageous occurrence were conducted by forming an aperture using two stainless steel plates, with the spacing between the two being varied to give aperture sizes, and thus beam widths (*D*), of 1-5mm. The laser power was fixed at 2 kW with unmodified beam dimensions of 6 x 10mm and traverse speeds ranging from 180-660 mm/min. Such an arrangement consequently allowed the examination of the effect of the laser beam width on the melt pool dimensions at constant laser power density and interaction time.

As is evident from Fig. 8, when the laser power density and traverse speed were fixed, the maximum melt depth was significantly affected by the aperture size. Indeed, if one considers the instance when the laser power was 2 kW and the traverse speed 540 mm/min, it is clearly evident that the change in aperture size from 1mm to 5mm resulted in a considerable difference in the maximum melt depth from 0.3mm to 1.2mm respectively. Such findings are in accord with those of Liu et al. [26], who noted that laser alloying melt depth of Ni on an Al-bronze substrate was affected significantly by aperture size.

7. Discussion

As the results of the mechanical and chemical tests show, the OPC glaze out performed the untreated OPC surface in almost all the test areas. Moreover, the generally superior mechanical and chemical performance of the OPC glaze over untreated OPC suggests that the life characteristics of the OPC glaze may also be superior to those of untreated OPC. This was especially true in the case of chemical resistance and water permeability, where the OPC glaze proved to be resistant to both. This marked variation in corrosion resistance and permeability performance is due to the difference in structure of the OPC glaze and the untreated OPC. Whereas the OPC glaze is of an amorphous nature, the untreated OPC is comprised of a porous polycrystalline structure, thus the untreated OPC is readily attacked by acids whilst the amorphous structure of the OPC glaze ensure an increase in acid resistance [27]. However, in any analysis of the wear life of the two materials the in-situ relative thickness of the OPC glaze and the untreated OPC surface of the concrete must be considered in order to give a true interpretation of the actual life characteristics, particularly when considering the wear resistance (with and without exposure to corrosive chemical agents). Consequently the increase in wear life can be given by

$$\text{Increase in wear life} = \frac{\text{OPC laser glaze wear life}}{\text{Untreated OPC wear life}} \qquad (2)$$

where,

$$\text{Wear life} = \frac{\text{Density . Thickness }(\text{mg.cm}^{-3}\text{.cm})}{\text{Wear rate }(\text{mg.cm}^{-2}\text{.h}^{-1})} \qquad (2a)$$

Table 1 summarises the wear rate details and the nominal life increase of the OPC glaze over the untreated OPC surface. As Table 1 shows, the OPC glaze gives an increase in actual life over the untreated OPC surface regardless of the environment. However, as one can see, the increase in actual life of the OPC glaze over the untreated OPC surface varies considerably depending upon the working environment. But, notwithstanding this, arguably the most common working environment for an OPC surface would involve some contact with at least detergent acids, therefore yielding significant economic savings since a HPDL glazed OPC surface lasts around 2.5 times longer than one which is unglazed.

When using identical laser operating parameters, remarkable variations in the depth of melting were observed for different beam widths. The principal factor effecting these differences is believed to be the marked differences in the temperature field distributions induced by the variations in the beam widths. It is generally understood that within a typical laser generated melt pool, the surface temperature is at a maximum in the centre, and decreases at a certain rate moving out from the centre to the edges thus establishing a temperature gradient [28, 29]. Clearly, such a condition naturally translates to a higher temperature in the centre of the melt pool throughout the melt pool section. Therefore melting to a greater depth will inherently occur in the centre of the irradiated region.

Consequently, this occurrence will be compounded as the width of the beam, and hence the generated laser melt pool, increases (assuming the laser parameters are the same). This is due to the fact that the temperature produced by a wide beam is always higher than that produced by a narrow beam as a result of the three-dimensional nature of heat transfer [26, 30]. Accordingly, the melt depth for a larger beam aperture size will always be greater than that for a narrow beam aperture size.

8. Conclusions

The use of a high power diode laser (HPDL) beam to produce a tough, inexpensive glaze which is amorphous on ordinary Portland cement (OPC) has been demonstrated successfully. Life assessment testing revealed that the OPC glaze had an increase in wear life of 1.3 to 14.8 times over an untreated OPC surface, depending upon the corrosive environment. Mechanical testing of the seals revealed that the average rupture strength of the OPC glaze was only 0.8 J, whilst the rupture strength of the untreated OPC surface was some 4.3 J. The average bond strength of the glaze was recorded as 24 MPa. This compares with 63 MPa for the untreated surface of the OPC. The maximum coverage rate that it may be possible to achieve using the HPDL was calculated as being 1.94 m^2/h. Moreover, the attendant problems of severe cracking and spallation which have been reported by many other workers when glazing with a CO_2 laser were not observed when using the HPDL. Variations in the width of the HPDL beam were seen to have a considerable affect on the melt depth. This is believed to be because the temperature produced by a wide beam is always higher than that of a narrow beam due to the three-dimensional nature of heat transfer.

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Fig. 1

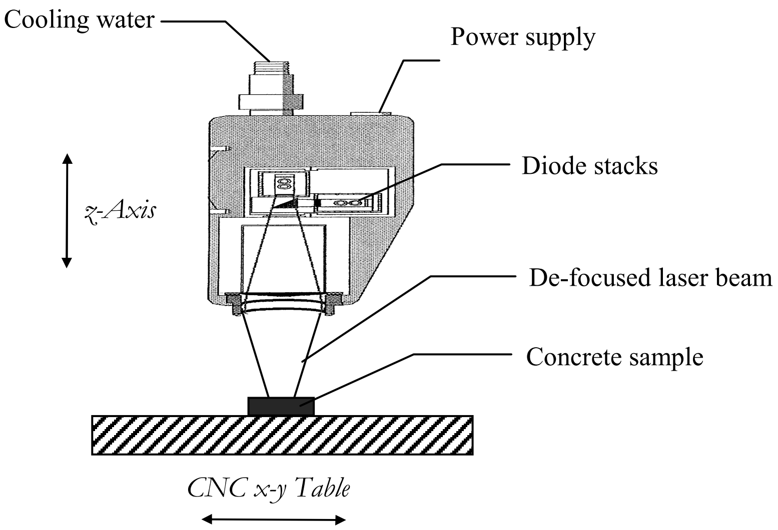

Cooling water
Power supply
Diode stacks
z-Axis
De-focused laser beam
Concrete sample
CNC x-y Table

Fig. 2

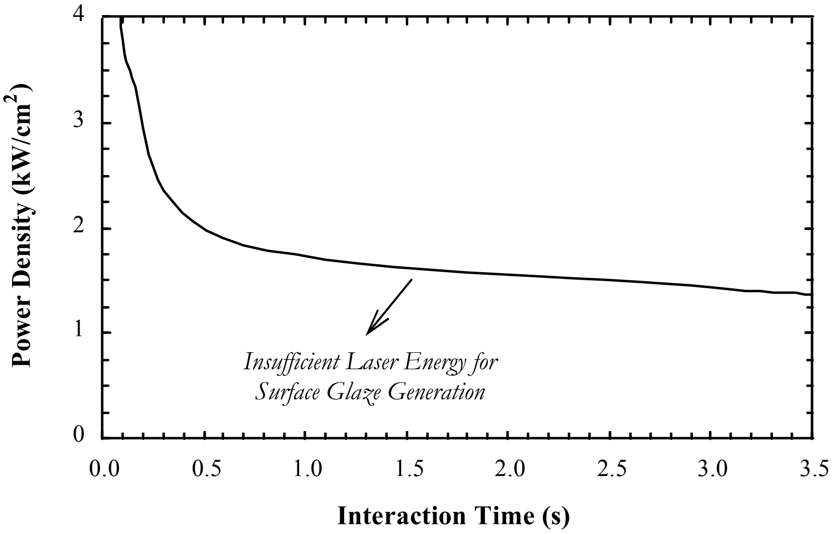

Power Density (kW/cm^2)
0
1
2
3
4
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
Interaction Time (s)
Insufficient Laser Energy for
Surface Glaze Generation

Fig. 3

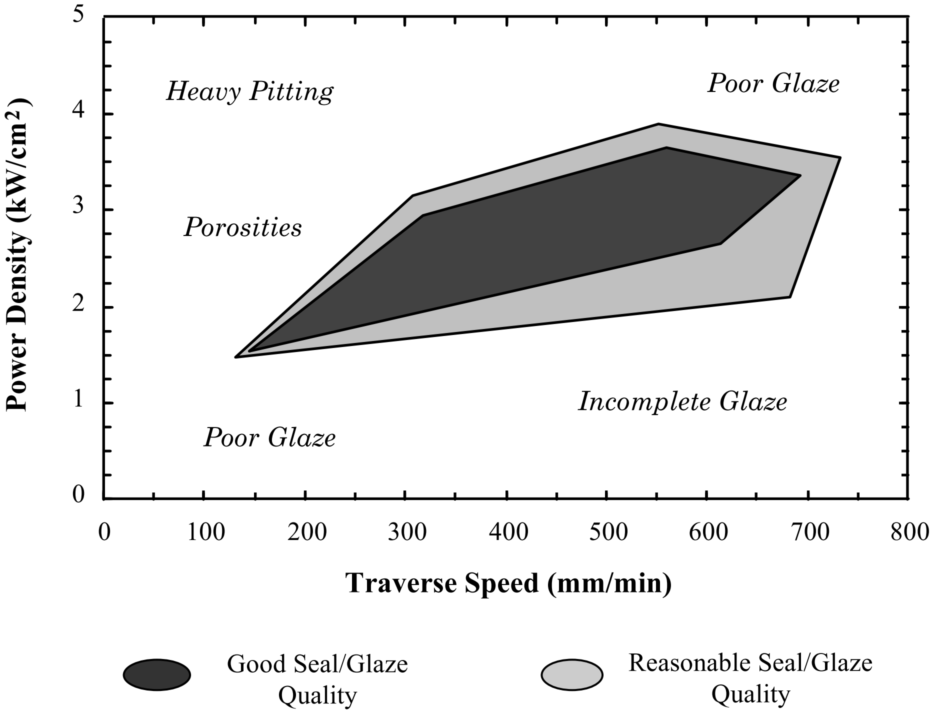

5
4
3
2
1
0
Power Density (kW/cm²)
0
100
200
300
400
500
600
700
800
Traverse Speed (mm/min)
Heavy Pitting
Poor Glaze
Porosities
Incomplete Glaze
Poor Glaze
Good Seal/Glaze Quality
Reasonable Seal/Glaze Quality

Fig. 4

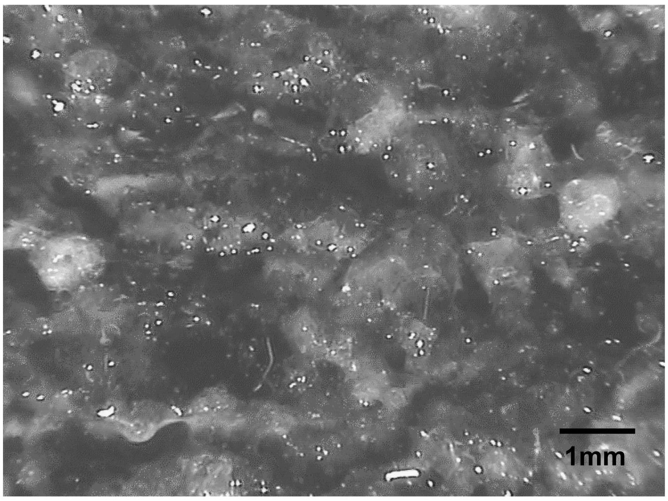
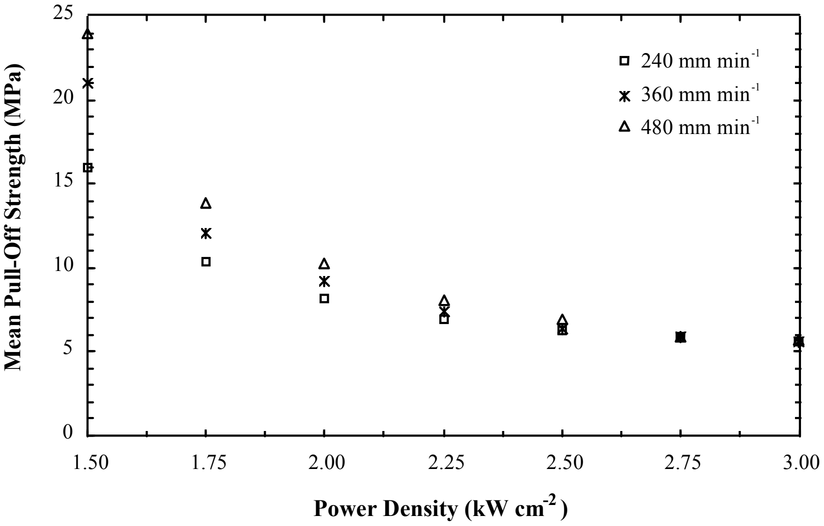


Fig. 5



Mean Pull-Off Strength (MPa)
0
5
10
15
20
25
1.50
1.75
2.00
2.25
2.50
2.75
3.00
Power Density (kW cm^{-2})
□ 240 mm min^{-1}
✱ 360 mm min^{-1}
△ 480 mm min^{-1}

Fig. 6

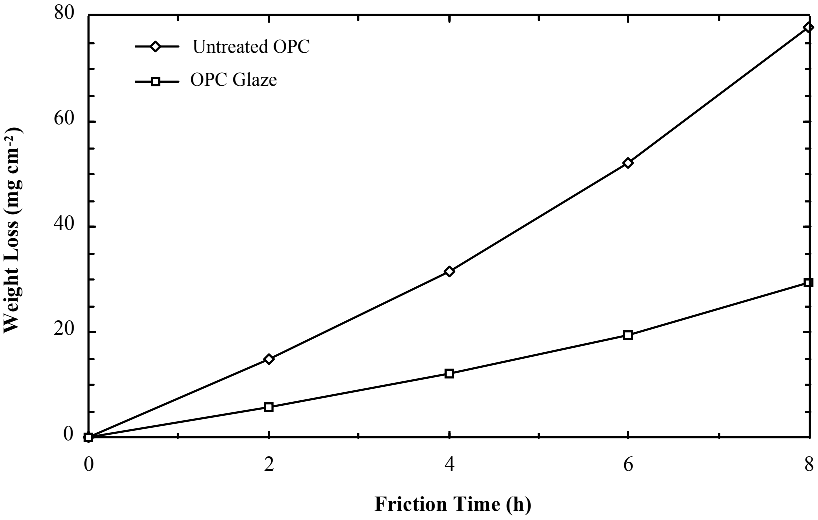
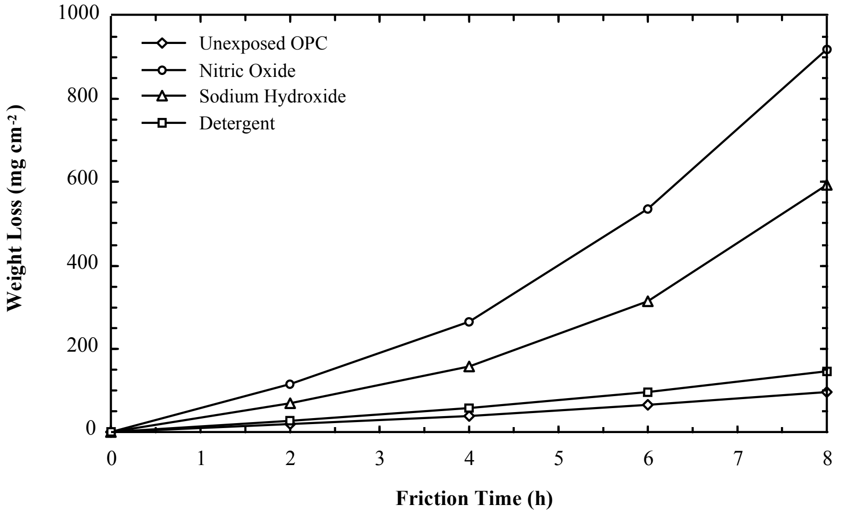
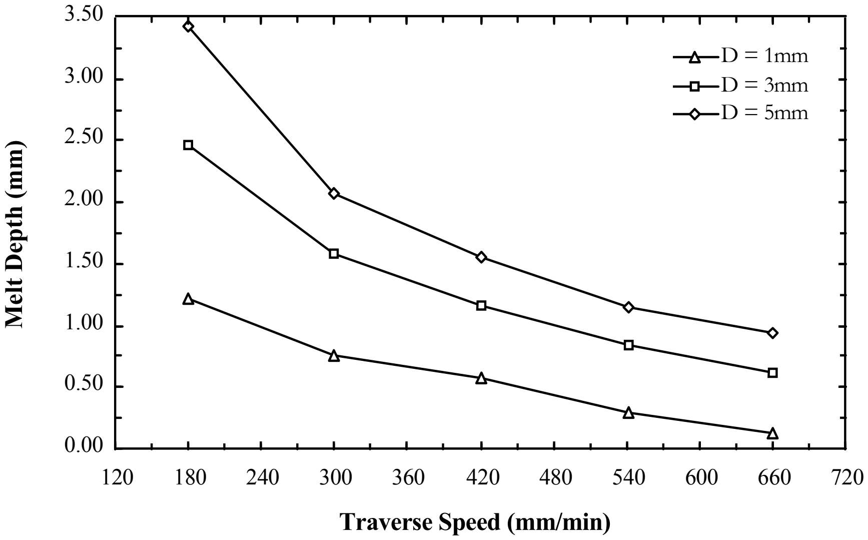

Untreated OPC
OPC Glaze
Weight Loss (mg cm^{-2})
Friction Time (h)
0
20
40
60
80
0
2
4
6
8

Fig. 7



Weight Loss (mg cm^{-2})
Friction Time (h)
0
200
400
600
800
1000
0
1
2
3
4
5
6
7
8
Unexposed OPC
Nitric Oxide
Sodium Hydroxide
Detergent

Fig. 8


Melt Depth (mm)
3.50
3.00
2.50
2.00
1.50
1.00
0.50
0.00
120
180
240
300
360
420
480
540
600
660
720
Traverse Speed (mm/min)
D = 1mm
D = 3mm
D = 5mm

List of Tables

Table 1

			Wear Rate ($mg.cm^{-2}.h^{-1}$)			
	Density	**Thickness**	**Unexposed**	**Detergent**	**NaOH**	**HNO_3**
Untreated OPC	2220 (kg/m^3)	1500 (μm)	9.8	18.5	73.8	114.8
OPC Laser Glaze	2000 (kg/m^3)	750 (μm)	3.5	3.5	3.5	3.5
Increase in Wear Life	~	~	**1.3**	**2.4**	**9.5**	**14.8**