Construction and Building Materials, Vol. 170, 747-756, 2018.

2 GROUT-CONCRETE INTERFACE BOND PERFORMANCE: EFFECT OF 3 INTERFACE MOISTURE ON THE TENSILE BOND STRENGTH AND GROUT 4 MICROSTRUCTURE

5 De la Varga I.¹, Muñoz J.F.¹, Bentz D.P.², Spragg R.P.¹, Stutzman P.E.², Graybeal B.A³

⁶ ¹ SES Group & Associates at Turner-Fairbank Highway Research Center, 6300 Georgetown

7 Pike, McLean, VA, 22101 (USA).

² National Institute of Standards and Technology, 100 Bureau Drive, Stop 8615 Gaithersburg,
 MD 20899-8615 (USA).

³ Federal Highway Administration at Turner-Fairbank Highway Research Center, 6300

11 Georgetown Pike, McLean, VA, 22101 (USA).

12 ABSTRACT

Bond between two cementitious materials is crucial in applications such as repairs, overlays, and 13 connections of prefabricated bridge elements (PBEs), to name just a few. It is the latter that has 14 special interest to the authors of this paper. After performing a dimensional stability study on 15 grout-like materials commonly used as connections between PBEs, it was observed that the so-16 called 'non-shrink' cementitious grouts showed a considerable amount of early-age shrinkage. 17 This might have negative effects on the integrity of the structure, due not only to the grout 18 material's early degradation, but also to a possible loss of bond between the grout and the 19 20 prefabricated concrete element. Many factors affect the bond strength between two cementitious materials (e.g., grout-concrete), the presence of moisture at the existing concrete substrate 21 surface being one of them. In this regard, pre-moistening the concrete substrate surface prior to 22 the application of the grout material is sometimes recommended for bond enhancement. This 23 24 topic has been the focus of numerous research studies in the past; however, there is still controversy among practitioners on the real benefits that this practice might provide. This paper 25 evaluates the tensile bond performance of two non-shrink cementitious grouts applied to the 26 exposed aggregate surface of a concrete substrate, and how the supply of moisture at the grout-27 28 concrete interface affects the bond strength. "Pull-off" bond results show increased tensile bond strength when the concrete surface is pre-moistened. Reasons to explain the observed increased 29 bond strength are given after a careful microstructural analysis of the grout-concrete interface. 30 Interfaces where sufficient moisture is provided to the concrete substrate such that moisture 31 movement from the grout is prevented show reduced porosity and increased hydration on the 32 33 grout side of the interface, which is thought to directly contribute to the increased tensile bond strength. 34

35 KEYWORDS

Cementitious grout; Interface moisture; Porosity; Tensile bond strength.

37 BACKGROUND

The bond between cementitious materials is a topic that has been extensively researched in the past decades [1–8]. The literature has identified a number of key factors that influences the measured bond strength, including the substrate surface preparation, the use of bonding agents, the mechanical properties of the two materials, and even the test method used to assess the bond strength [1,5,9].

One parameter that is recognized to affect the bond performance between two cementitious 43 44 materials is the availability of moisture at the concrete substrate surface prior to the casting of the new material [10-12]. It has been reported that when pouring a fresh material over a dry concrete 45 substrate, the substrate may absorb part of the mixing water from the former, thus forcing the 46 water to migrate from the new material to the substrate [13]. This effect might be more 47 pronounced in highly fluid materials (e.g., cementitious grouts). The water migration would not 48 49 only lead to internal stresses at the interface, but could also reduce the strength of the grout material due to a lack of sufficient water for its hydration. It is therefore believed that by 50 providing extra moisture at the interface prior to the application of the fresh material, it is 51 possible to reduce the water migration. This has been observed using neutron images [14] in 52 53 repair mortar overlays cast over concrete substrates in "saturated surface dried" (SSD) conditions, compared to dry substrates. 54

While there is a certain degree of consensus regarding the beneficial effects of pre-moistening the substrate, there is some controversy among researchers and practitioners as to if this is really the case [11,12]. Courard, et al., have reported that there is an optimum substrate moisture range between 55 % and 90 % degrees of saturation [8]. Outside of this range of degree of saturation, the bond strength decreases. The practice of pre-moistening the concrete substrate surface in order to achieve SSD conditions has become common in the construction industry [15].

61 In a review of the literature of the topic, the authors have identified that recommendations to pre-62 moisten the substrate are dependent on the test method being used, the surface preparation techniques used, and the types of materials being bonded. This study focuses on evaluating the 63 effect that the supply of extra moisture at a grout-concrete interface has on the bond 64 65 performance, specifically targeting the potential application in connections of pre-fabricated bridge elements (PBE). For that purpose, the surface of the concrete substrate has been prepared 66 according to current field practices (more details will be given in the next sections). This study 67 assesses the tensile bond strength between cementitious grouts and a concrete substrate using 68 "pull-off" bond tests. Additionally, the paper presents scanning electron microscope (SEM) 69 images along with measured porosity and hydration profiles in the grout material along the 70 71 interface with the concrete material to correlate the pull-off bond results to the grout 72 microstructure features.

73

75 MATERIALS

76 *Concrete*

An ordinary portland cement, ASTM C150-16 Type I/II, with a Blaine fineness of 382 m²/kg, 77 and a density of 3070 kg/m³, was used to prepare the concrete. The fine aggregate (FA) used was 78 ordinary river sand with an apparent specific gravity of 2.59. The coarse aggregate (CA) 79 consisted of dolomitic limestone with an apparent specific gravity of 2.85. The concrete mixture 80 was developed to perform similarly to a prefabricated concrete element in terms of strength. 81 82 Therefore, the concrete was designed with a water-to-cement ratio (w/c) of 0.35 by mass, 83 cement:FA:CA ratio of 1:1.7:2.5 (by mass), a minimum slump of 76 mm (3 in.) (achieved by 84 using a high-range water reducer), and a targeted 28-d compressive strength of 55 MPa (8000 psi). 85

86 'Non-Shrink' Cementitious Grouts

Two commercially-available 'non-shrink' cementitious grouts were used in the study, labelled here as Grout A and Grout B. The grouts are supplied in a bag containing the solid fraction (e.g., cementitious materials, additives, and fine aggregates) that is mixed with a certain amount of water following the manufacturer's recommendations to obtain an average flow of 100 % per ASTM C1437. Grout A requires a water-to-solids ratio (*w/s*) of 0.16 by mass and produces a 28-d compressive strength of 62 MPa (9000 psi), whereas grout B requires a *w/s* of 0.17 and provides a 28-d compressive strength of 48 MPa (7000 psi).

94 EXPERIMENTAL

95 Grout-Concrete Specimen Preparation

A concrete slab with dimensions of 914 mm x 914 mm x 102 mm (36 in. x 36 in. x 4 in.) was 96 97 used as the substrate for the pull-off bond tests. The concrete slab was cured for 14 d after 98 casting at a temperature of 23 °C \pm 1 °C (73.4 °F \pm 1.8 °F) and relative humidity of 50 % \pm 5 %. 99 (14 d was selected for convenience; the authors consider this age to be representative of the substrate age at the moment of the grout pour. 14 d curing was sufficient to provide a concrete 100 tensile strength that was greater than that of the grout-concrete interface). The top surface of the 101 concrete slab was pressure washed at 24 h after casting in order to create an exposed coarse 102 103 aggregate interface (see Figure 1(a)), facilitated by using a commercially available in-form paintlike retarding agent. This is becoming a common substrate surface preparation method in several 104 states for PBE connection applications [16], and it is the reason why it has been selected for this 105 106 study.

107 A 50-mm (2-in.) thick grout overlay was cast over the top surface of the concrete slab so that an 108 interface between the two materials is created. Moisture at the grout-concrete interface was 109 provided by ponding the exposed aggregate concrete surface with water during the 24 h period

- that preceded the casting of the grout, and manually drying the surface with paper towels, so that
- an SSD condition was achieved. Therefore, two different types of specimens were prepared: 1)
- "control" (with no moisture added to the interface), and 2) "SSD" (with moisture added to the
- 113 interface).
- 114



Figure 1. (a) Grout-concrete slab for pull-off tests (concrete substrate surface was prepared with
an in-form retarder agent to expose the coarse aggregates, as shown in the figure) with
illustration of pull-off bond test method via ASTM C1583, and (b) four possible failure modes
obtained after executing pull-off tests: (1) substrate failure, (2), interface failure, (3) grout
failure, (4) glue failure.

120 *Pull-off bond tests*

The bond assessment was performed according to the ASTM C1583 test method (direct tension "pull-off" test) on the grout-concrete slab (Figure 1(a)). In this test, a 50-mm (2-in) diameter steel disc is glued on the top surface of the grout. The test specimen is formed by partially drilling a core perpendicular to the surface, and penetrating down into the concrete material, approximately 25 mm (1 inch) below the grout-concrete interface. A tensile load is applied to the steel disc at a constant rate of 35 kPa/s \pm 15 kPa/s (5 psi/s \pm 2 psi/s) until failure occurs. The failure load and the failure mode were recorded and the nominal tensile stress could thus be calculated. If failure occurred at the grout-concrete interface, then the true bond strength could be assessed. If failure occurs in either the concrete substrate or grout material, then the tensile strength of the failing material could be assessed and the interface bond strength could be recognized to be higher than the value achieved. Finally, the test was rejected if failure occurred at the glue-grout interface (Figure 1(b)). According to the ASTM test method, at least three valid tests should be completed and the results averaged for any particular failure mode.

134 *Microstructural analysis*

After completion of the pull-off bond tests, grout-concrete specimens were prepared for microstructural analysis. Cores 50-mm (2-in.) in diameter were extracted from the grout-concrete slab and cut in half to expose the interface (Figure 2). The specimens were then immersed in isopropanol for 48 h, dried in a vacuum oven at 25 °C (77 °F), impregnated with a low-viscosity epoxy resin, lapped, and polished to a surface roughness of 0.25 μ m. All the cuts were performed

140 using inorganic oil as the lubricant.

141



Figure 2. (a) Cylindrical cores extracted from grout-concrete slab, (b) grout-concrete specimen
 prepared for SEM/EDS analysis.

The microstructure of the polished specimens was examined using a FEI Quanta 650¹ scanning 144 electron microscope (SEM) equipped with a concentric backscatter detector coupled with Energy 145 146 Dispersive X-ray Spectroscopy (EDS/EDX). The microscope was operated under high vacuum at 15 KV and with a 10-mm working distance. Large backscatter electron (BSE) and EDS/EDX 147 mapping areas were collected using Aztec 2.4 EDS microanalysis software. The maps had a total 148 of 299 fields, each one with a 3000x magnification, which covered a total area of 1.79 mm² 149 (0.0028 in²). An example of a large BSE map for a grout-exposed coarse aggregate interface is 150 151 displayed in Figure 3(a). A minimum of six maps per specimen were collected in random areas,

¹ Certain commercial equipment and software are identified to describe the subject adequately. Such identification does not imply recommendation or endorsement by FHWA or NIST, nor does it imply that the equipment identified is necessarily the best available for the purpose.

- covering a total area of approximately 9 mm² (0.014 in²) of the interface, to ensure that the analysis was representative of each sample. The area in the large BSE maps corresponding to sand particles in the grout was removed using an image treatment process based on the EDS elemental map of calcium. Specific details of the methodology can be found in Beyene et al. [17]. The obtained map of sand particles in the grout was then subtracted from the original large BSE map to obtain the final large paste map, used for the porosity measurements, as illustrated in Figure 3(b).
- The porosity of the final large paste map, as shown in Figure 3(b), was evaluated on the grout paste adjacent to the surface of the concrete, and up to 100 μ m away from the latter. A porosity profile was obtained by extracting consecutive 10- μ m wide bands, in a similar process to that proposed elsewhere [18,19] to characterize the interfacial transition zone around aggregates in concrete. Examples of this segmentation process and a typical 10- μ m wide band are displayed in Figure 3(c) and (d), respectively.

The image analysis software ImageJ $1.49v^1$ was used for quantification of the porosity in each of 165 the BSE 10-µm wide bands. Specific details of the process to measure porosity are described in 166 Beyene et al. [17]. It is important to mention that the pixel size in the images (1 pixel 167 representing 0.2 µm x 0.2 µm) allowed for the quantification of pores ranging from 0.45 µm 168 to 40 µm, herein referred to as capillary pores. The upper limit of 40 µm was selected based on 169 the maximum pore size observed in these types of interfaces. The lower limit was selected based 170 171 on the Nyquist theorem [20]. The large BSE maps displayed in this paper are representative of the entire samples. The coefficient of variation (CoV) of measured porosity on each of 172 the 10-um wide bands evaluated was found to be less than 8.5×10^{-4} %. 173

174

175

176

177

178



Figure 3. Example of segmentation process based on calcium EDS mapping used to study the
 microstructure of the grout-concrete interface. The process is illustrated with a large map of a
 control sample cured for a total of 14 d on the specific grout-exposed aggregate region: (a)
 original large BSE map, (b) large paste map after removal of grout sand particles, (c) location
 along the interface of the 10-µm wide bands, and (d) example of a 10-µm wide band extracted
 20 µm away from the exposed aggregate surface.

187 **RESULTS**

188 Pull-off bond tests

Table 1 shows the pull-off bond strength results obtained for various batches of the two 189 190 cementitious grouts used in the study and applied to an exposed aggregate concrete substrate 191 surface. The results were collected over a period of about two years, and the variability in the 192 results observed within Grout A are attributed to potential modifications in the product formulation made by the manufacturer during this time. Results labelled as "control" correspond 193 194 to the specimens where no moisture was added (i.e., specimens were maintained at drying conditions in a room that holds the temperature and relative humidity at 23 °C \pm 1 °C (73.4 °F \pm 195 1.8 °F) and 50 % \pm 5 %, respectively). "SSD" corresponds to the specimens where additional 196 moisture was provided via 24-h water ponding and subsequent paper towel drying. All the 197 specimens failed at the grout-concrete interface, so the (theoretically) true bond strength was 198 199 assessed. In all cases, the bond strength increased when an SSD condition was provided on the concrete surface, doubling the bond strength with respect to the control in some cases 200 201 (percentage increase is shown in the table). In five out of the nine cases presented, the bond strength mean values for the two surface conditions (SSD and control) were statistically different 202 at the 0.05 significance level. 203

204 205

Table 1. Results of direct tension (pull-off) testing (Control vs. SSD) for various batches of two
 cementitious grouts throughout the period of 2 years.

| Material | Batch | Grout Age at Test (d) | Control (MPa) | SSD (MPa) | Control (psi) | SSD (psi) | Bond Strength Increase (%) | t0.05 |
|----------|-------|--------------------------------|------------------|-----------------|------------------|--------------|-------------------------------------|-------|
| Grout A | 1 | 2 | 1.90 ± 0.40 | 2.68 ± 0.44 | 276 ± 57 | 389 ± 64 | 41 | Ν |
| | | 14 | 2.77 ± 0.25 | 3.46 ± 0.50 | 402 ± 37 | 501 ± 72 | 25 | Y |
| | 2 | 14 | 2.37 ± 0.16 | 2.73 ± 0.61 | 343 ± 23 | 397 ± 88 | 16 | Ν |
| | 3 | 14 | 2.67 ± 0.55 | 2.81 ± 0.14 | 387 ± 80 | 408 ± 21 | 5 | Ν |
| | 4 | 7 | 1.79 ± 0.67 | 3.70 ± 0.26 | 259 ± 98 | 536 ± 38 | 107 | Y |
| | 5 | 14 | 3.78 ± 0.36 | 4.50 ± 0.23 | 549 ± 52 | 652 ± 34 | 19 | Ν |
| | 6 | 14 | 1.49 ± 0.23 | 3.04 ± 0.56 | 216 ± 34 | 441 ± 82 | 104 | Y |
| Grout B | 1 | 2 | 1.82 ± 0.12 | 2.74 ± 0.42 | 263 ± 18 | 398 ± 62 | 51 | Y |
| | | 14 | 2.06 ± 0.57 | 3.18 ± 0.36 | 300 ± 82 | 461 ± 52 | 54 | Y |

207

208 *Microstructural analysis*

The microstructural analysis of the grout-concrete interface (hereafter referred to as just "interface") was performed only on some grout-concrete specimens collected from the pull-off tests using grout A (see highlighted cases in Table 1). This material was chosen over grout B, since the latter contained a specific component in its original formulation that triggered a gas reaction during the curing process. Evidences of this gas reaction in grout B were observed by the presence of uniformly distributed air bubbles ranging from 50 µm to 150 µm during a preliminary SEM analysis of fresh fracture surfaces of the interface. (Some cementitious grouts are designed to counteract later age hydration-related shrinkage by adding expansive agents, including some that generate gases). As such, in order to eliminate the gas formation as an additional variable that might have an effect of the final interface microstructure, only grout A was selected for further microstructural investigation.

The microstructural analysis presented in this paper corresponds only to the grout side of the 220 221 interface. It is therefore assumed that the microstructural characteristics of the substrate in both "control" and "SSD" conditions would be very similar. This was in fact confirmed by an SEM 222 223 porosity analysis of thin sections prepared from concrete specimens extracted prior to the grout pour. Despite submerging the concrete surface during 24 h to achieve the SSD moisture 224 condition (which might further hydrate the paste at the concrete surface), no significant 225 differences in the measured porosity were observed between the control and SSD concrete 226 227 specimens. The porosity values in both the control and SSD concrete ranged from 0.60 % to 0.65% in the areas close to the concrete surface (from 0 µm to 20 µm of distance) down to 0.20 % in 228 229 those areas farther away (from 90 µm to 100 µm). The percent porosities were within 0.05 of one 230 another for the two conditions.

231 Interfaces are commonly seen as lines (in 2D images) or planes (in 3D images) between two different phases (e.g., materials). This is easy to observe when the two phases (or materials) are 232 233 different in terms of, for instance, porosity (e.g., aggregate-paste interface). However, this approach proves to be difficult for interfaces formed between cementitious materials, especially 234 in paste-to-paste interfaces, where both materials show similar microstructures in terms of 235 porosity. As such, in this paper an interface was considered more as a region (that is, a plane 236 in 2D images, or a volume in 3D images), including areas that extend several microns within the 237 238 two interfacial materials. It is also worth mentioning that heterogeneity is one of the principal 239 attributes of an interface at a micro-scale level. This further complicated the possibility of 240 obtaining representative porosity measurements even though large maps were used in the evaluation. Even so, two different areas could be distinguished at the interface since the grout 241 was poured over a concrete surface where the coarse aggregate was previously exposed. The first 242 area corresponded to that where the grout is in direct contact with the exposed coarse aggregate 243 of the concrete material (G-A interface), while the second area represented locations where the 244 grout is in direct contact with the paste fraction of the concrete material (G-P interface). This 245 246 latter area is typically confined between exposed coarse aggregate particles, creating a "valleylike" configuration with the paste at the bottom. An illustration of G-A and G-P interfaces is 247 248 shown in Figure 4. As already mentioned only the grout side of the interface was analyzed in this 249 paper.





Figure 4. Illustration of G-A and G-P interfaces in a grout-concrete interface with exposed coarse aggregate on the concrete side. Note: the illustration is not to scale.

254 Examples of representative large maps of G-A interfaces are displayed in Figures 3(a) and 5. As observed, the resulting microstructure of a G-A interface is characterized by an "unconfined" 255 configuration, which makes it easy for the grout to consolidate. The "unconfined" microstructure 256 257 contributed to minimize the commonly-known "wall effect" [18,21-23], which would reduce the 258 packing efficiency of the grout particles at the vicinity of the exposed coarse aggregate surface. 259 The consequence of this "unconfined" structure was a dense microstructure at the interface with a reduced porosity in the grout. However, local disturbances in the packing efficiency that 260 261 created "pockets" of porosity were common in this type of interface. These porosity "pockets" were caused by the proximity of sand particles and/or air voids to the exposed coarse aggregate 262 surfaces, as well as the confluence of several sand particles at the interface region. Examples of 263 these localized disturbances in the packing efficiency are depicted in Figure 5 as Area A (for 264 265 sand particles) and Area B (for air voids). It is important to mention that "porosity pockets" caused by the proximity of sand particles can also be found in bulk areas of the sample, a few 266 hundreds of microns away from the exposed coarse aggregate surface (Area C in Figure 5). 267 However, the influence of these high porosity areas in the bulk was disregarded in the porosity 268 269 evaluation presented in this paper by limiting the area of interest to being within 100 µm of the exposed coarse aggregate surfaces (G-A interfaces) or concrete paste surfaces (G-P interfaces). 270



Figure 5. Large BSE map of a G-A interface of a control (non-SSD) sample cured for 2 d. It
shows an "unconfined" configuration that facilitated the packing of the grout particles on top of
the exposed coarse aggregate surface. Local porosity was created at the interface by the presence
of sand particles of the grout (Area A) or air voids (Area B). The same phenomenon was
observed in the bulk (Area C).

When quantifying the amount of porosity in G-A interfaces, it is observed that the presence of 278 279 sand particles has an influence on the total porosity. This is illustrated in Figure 6, where sudden increases in the porosity fraction (indicated by dashed arrows) occurred as we approach the 280 exposed coarse aggregate surface in locations where sand particles were present. In other words, 281 282 porosity tended to increase in between the sand and exposed coarse aggregate particles, especially near sand particles. This effect was observed regardless of the curing time (2 d 283 or 14 d) and the type of sample (control or SSD). Therefore, SSD conditions did not have any 284 effect on the microstructure of G-A interfaces, at least in terms of the measured porosity. 285

286 Contrary to G-A interfaces, the microstructure of G-P interfaces was highly influenced by a 287 "confined" configuration, due mainly to the proximity of exposed coarse aggregates from the 288 concrete material (previously expressed as "valley"-like regions). The configuration caused a 289 poor packing efficiency of the grout in these regions, as reflected by the large porosity observed 290 in Figure 7. The presence of large sand particles in such a confined environment actively 291 contributed to further increase the porosity. This resulted in the presence of pores with sizes up 292 to $40 \mu m$.





Figure 7. Large BSE map of a G-P interface of a control (non-SSD) sample cured for 2 d. It
 shows a "confined" configuration that impaired proper packing of the grout.

Quantification of the overall porosity distribution of the grout as a function of distance from the 302 concrete surface (either exposed aggregate or paste surfaces) is presented in Figure 8. The results 303 include both types of interfaces (G-A and G-P) for the two types of tested specimens (control and 304 305 SSD) and cured for 2 d and 14 d. Several general observations can be made from the results. First, all the tested samples showed significant porosity values within the analyzed 100 µm band. 306 The values ranged from about 10 % to 30% in the areas close to the concrete surface (from 0 µm 307 to 20 µm of distance), down to approximately 5 % in those areas farther away (about 90 µm 308 to 100 µm). The values of porosity progressively decreased in moving away from the concrete 309 surface. This tendency was occasionally disrupted, causing a peak in porosity, due to the 310 proximity of sand particles (as previously explained in the paper). These peaks in porosity were 311 mostly a characteristic of the G-A interfaces as shown in Figure 8 for the SSD specimen at 14 d. 312



313

Figure 8. Overall porosity distribution of the grout (including both G-A and G-P interfaces). (These plots are based on averaged measurements of three representative areas. While the CoV

was below 15 % in the 0 to 20 μ m bands, this value is less homogeneous in the rest of the bands, achieving values of 50 %. This high variability is caused by the presence of sand particles near the interface, as shown in figure 6).

319

320 When comparing the porosity distribution in the two types of interfaces studied (G-A vs. G-P), it can be observed that for G-A interfaces, the maximum porosity within the first 20 µm was 321 approximately between 10 % and 15 %. This number slightly decreased to 5 % to 10 % by the 322 323 end of the 100 µm band. The increase of grout curing time from 2 d to 14 d did not cause a 324 significant reduction in the measured porosity (between $0.2 \,\mu m$ to 40 μm pore size) regardless of the type of specimen, control or SSD. On the other hand, porosity values in G-P interfaces were 325 326 not significantly affected by the increase in curing time; however, the values were influenced by the type of specimens. In general, porosity in the first 20 µm band of G-P interfaces was higher 327 for the control (around 22 % and 30 % at 2 d and 14 d, respectively) than for SSD specimens 328 (18 % and 13 % at 2 d and 14 d, respectively). The reduction in porosity in the SSD specimen 329 with respect to the control is indicated by the arrows in Figure 8. A closer comparison of the pore 330 size distribution of each band within the first 40 µm for the 14-d G-P interfaces in the control 331

and SSD specimens provides additional information, as illustrated in Figure 9. The higher overall porosity in the control with respect to the SSD specimen was caused by a higher number of larger capillary pores ($25 \mu m$ to $40 \mu m$ in size) at 10 μm and 20 μm distances from the concrete paste surface. At larger distances from the surface, both specimens had similar porosity size distributions.





338

341

Figure 9. Comparison of pore size distribution analysis in 14-day G-P interfaces between the control and SSD specimens

The difference in overall porosity and pore size distribution in the first 20 µm at G-P interfaces 342 can be attributed to one of the two following mechanisms. The first mechanism assumes that the 343 344 presence of water in the capillary pores of the concrete paste in the SSD sample did help in reducing the higher porosity and refining the pore size distribution by maximizing the formation 345 of hydration products at the grout side of the interface. Theoretically, neither unhydrated solids 346 nor porosity will contribute much to bonding. For this reason, the amount of hydration products 347 was measured on G-P interfaces in the 14 d specimens (both Control and SSD). Figure 10 shows 348 that the amount of hydration products in the SSD specimen is significantly higher at the 0.05 349 350 significance level than that of the control specimen throughout the entire 100 µm band that was analyzed, and particularly in the 10 µm band adjacent to the existing substrate. The differences in 351 hydrated and unhydrated volume fractions between the control and SSD conditions are much 352 greater than their difference in porosity (Figure 8). 353







Figure 10. Overall porosity, hydrated, and unhydrated distribution on the grout side of the interface in 14 d G-P interfaces, for both control and SSD specimens. (These plots are based on averaged measurements of three representative areas. The CoV was below 15 %).

The second mechanism would consist of a moisture-air exchange occurring between the grout 359 and concrete materials. In other words, the air present in the concrete's capillary pores would 360 361 migrate into the grout as the moisture in the grout is absorbed by the concrete substrate. This has been observed by other researchers [13]. According to this mechanism, the migrated air should 362 remain as (circular) voids near the interface. The extent of this mechanism was determined by 363 364 analyzing the degree of circularity of the previously identified porosity in the first 40 µm at G-P interfaces. It was assumed that circularity values of air voids will be close to one; while 365 circularity values close to zero will be indicative of voids caused by packing deficiencies. The 366 results in the circularity index for both samples are shown in Figure 11. The overall low 367 circularity values of the porosity, close to 0, support that a poor consolidation of the grout onto 368 369 the exposed concrete aggregate surface was the main cause of the higher and coarser porosity in 370 the control specimens in this case.





Figure 11. Circularity index analysis of the porosity for the 14-day G-P interfaces of the control
 and SSD specimens.

375 **DISCUSSION**

372

The pull-off bond results obtained in this study show a clear effect of pre-moistening the concrete surface prior to the grout pour by using the SSD approach explained above. The tensile bond strength is increased by up to 107 % in some cases. These are significant increases that merit a closer look. As such, SEM images and porosity profiles of the grout along the interface have been used to better understand the mechanism behind the enhanced bond performance.

In the case presented here, the concrete surface has been treated with a retarder so that the coarse 381 382 aggregate fraction is exposed. This implies that the grout-concrete contact occurred in two 383 different types of surfaces: exposed coarse *aggregate* and concrete *paste* surfaces, respectively creating two types of interfaces named here as G-A and G-P. While G-A interfaces were 384 characterized by a more "unconfined" microstructure, G-P interfaces tended to be more 385 386 "confined", typically in between exposed coarse aggregate particles, forming "valley-like" regions. G-A interfaces occupied about 70 % of the entire concrete surface based on the mixture 387 design used in this study, and typically provide initial bond strength through mechanical 388 interlock, as shown elsewhere [24]. 389

Most of the SEM images collected showed grout packing deficiencies and increased local 390 porosity for the particular grout used in the study (grout A) in both G-A and G-P interface types 391 (the authors mainly attribute this to non-optimum gradation of sand and fillers in the grout 392 formulation). Even so, the grout tended to consolidate much better over G-A interfaces than over 393 394 G-P interfaces, due mainly to the more "unconfined" configuration of G-A interfaces as opposed to that of G-P interfaces (more "confined"). This resulted in a poor grout consolidation in G-P 395 interfaces, thus depicting a more porous type of microstructure. In addition to this, water 396 migration is more common at these G-P interfaces since water is moving from one porous 397 398 material (grout) into another porous material (concrete paste). The consequence of this is that the grout may not properly hydrate in the control sample (non-SSD) due to a lack of available water 399 400 needed for hydration. Therefore, it is expected that SSD conditions will be beneficial in this regard, especially in G-P interfaces. 401

When evaluating the effect that SSD conditions had on the amount of porosity found in each of 402 these interface types, it was found to have a different effect. In G-A interfaces, similar porosity 403 404 values (approximately 0.50 %) were found at the vicinity of the concrete surface regardless of the moisture condition (control or SSD). This makes sense as it is not expected that a film of free 405 water stays over the exposed coarse aggregate surface when applying the SSD condition (as 406 surfaces were manually-dried with paper towels). Therefore, it can be inferred that SSD 407 conditions did not really change the moisture conditions in G-A interfaces. On the other hand, 408 409 G-P interfaces showed clear differences in the measured porosity near the concrete paste surface (especially within the first 20 μ m), with reduced values in the SSD samples 410 (approximately 15%, compared to 30% in control samples at 14 d). As already mentioned, 411 water migration is more common at these G-P interfaces. For the control samples, as water is 412 413 absorbed by the substrate, the cement particles in the grout will be drawn into a more tightly packed (denser) configuration at the interface, as supported by the plots in Figure 10. Water 414 removal from the grout at this interface will also increase its viscosity (and yield stress), so that 415 the grout will not easily conform to the rough concrete surface and "pockets" of porosity (non-416 circular pores) will remain. Conversely, it is believed that SSD helps in preventing (or at least 417 reducing) this water (and particle) movement by keeping the capillary pores of the concrete 418 substrate "more saturated", while at the same time increasing the degree of hydration of the grout 419 (denoted by the reduced measured porosity and increased hydration products), as shown in 420 figure 10. The increased degree of hydration observed in SSD samples compared to the control 421 422 would imply not only improved mechanical properties of the grout (e.g., tensile strength), but also more hydration products in direct contact with the concrete surface. These two aspects 423 (better mechanical properties and higher degree of hydration) may explain the increased bond 424 strength observed in the SSD samples. 425

426 More hydration products forming in SSD samples would also mean that larger contact areas exist 427 between the grout and the concrete at the interface. This raises questions such as the type of 428 crystals forming when SSD conditions are provided, compared to those in the control. It is 429 believed that SSD conditions would not only increase the degree of hydration in G-P interfaces, but would also concurrently cause the formation of thicker (and probably stronger) crystal
structures that would provide larger contact areas with the concrete surface. This topic is being
further investigated. Additionally, less porous G-P interfaces should mean more durable bonds,
an important aspect when evaluating the long-term performance of the grout-concrete interface
that requires more research.

435 It has then been shown that SSD conditions have a major contribution in reducing the measured porosity and increasing the volume fraction of hydration products in G-P interfaces for the 436 437 materials examined in this study, thus increasing the tensile bond strength. This is especially evident in the type of exposed aggregate concrete substrate surface presented in this paper. 438 During the preparation of this paper, other pull-off bond results were collected on sandblasted 439 concrete surfaces, and different effects were observed (that is, SSD was not as beneficial as in an 440 441 exposed coarse aggregate surface). It is then conjectured that providing interface moisture is beneficial in interfaces where "valley-like" regions are present, as the moisture can improve the 442 consolidation, hydration and porosity properties of those regions. All this indicates that not only 443 444 the bond test method used but also the type of substrate surface preparation play an important role in the effect that interface moisture has on the bond performance. As such, it is important to 445 note that the conclusions made in this paper only apply to this type of grout-concrete interface 446 and for tensile pull-off testing, which was specifically chosen to target connections in precast 447 bridge elements. 448

449

450 **CONCLUSIONS**

This paper focuses on evaluating the tensile bond performance of cementitious grouts to an exposed aggregate concrete substrate surface by means of pull-off tests and consequent microstructural (i.e., porosity) characterization of the grout-concrete interface. Based on the results obtained, the following conclusions can be made:

455

For the case of coarse aggregate exposure, the presence of moisture on the concrete surface supplied in this case as SSD conditions prior to the grout placement has been demonstrated to have benefits in terms of enhanced tensile bond strength, achieving in some cases up to a 107 % increase compared to a control (dry) specimen. This contradicts some other research studies, such as the one presented by Beushausen et al. [12], where other bond test methods and/or substrate surface preparation were employed..

The use of a retarder to provide an exposed coarse aggregate type of surface on the concrete substrate leads to the formation of two types of interfaces: 1) grout-aggregate (G-A), and 2) grout-paste (G-P). The consolidation of the grout is favored on G-A interfaces, characterized by a more "unconfined" configuration, compared to that of G-P interfaces, shown to have a more "confined" configuration. The main consequence of this is higher porosity values

d67 observed in G-P interfaces as compared to G-A interfaces, particularly under dry surfaced68 conditions.

- The presence of moisture in the SSD specimens actively contributed to the reduction of the initial larger porosity observed in G-P interfaces, increasing the degree of hydration and the volume fraction of formed hydration products. It is then conjectured that this "densification/enhanced hydration" of G-P interfaces, together with the (very likely) stress reduction caused by the reduction of water migration shown elsewhere [14], is the main cause of the increased tensile bond strength observed in the pull-off tests.
- It has been shown that more hydration products are formed in G-P interfaces, directly contributing to the densification of the grout-concrete interface, and thus increasing the tensile bond strength.

Finally, the authors recommend pre-moistening the concrete substrate surface to enhance the tensile bond performance of a grout-exposed aggregate concrete element. This type of interface is common in PBE connection applications. Bond improvement might not be considerable, but is often significant. The provision of moisture at the interface could be a feasible means to improve

- tensile bond, among other available techniques (e.g., surface preparation, bonding agents, etc.).
- 483

484 ACKNOWLEDGEMENTS

The authors would like to thank Daniel Balcha and Michael Gregory of SES Group & Associates 485 for their technical assistance. The research which is the subject of this document was funded by 486 the U.S. Federal Highway Administration. This support is gratefully acknowledged. The 487 publication of this report does not necessarily indicate approval or endorsement of the findings, 488 opinions, conclusions, or recommendations either inferred or specifically expressed herein by the 489 Federal Highway Administration, the National Institute of Standards and Technology, or the 490 United States Government. The reviews of Dr. Scott Jones, Dr. Chiara Ferraris, and Dr. Ken 491 Snyder (NIST) and Dr. Jussara Tanesi (SES Group) are greatly appreciated. 492

493 **REFERENCES**

- 494 [1] A. Momayez, M.R. Ehsani, A.A. Ramezanianpour, H. Rajaie, Comparison of methods for
 495 evaluating bond strength between concrete substrate and repair materials, Cem. Concr.
 496 Res. 35 (2005) 748–757. doi:10.1016/j.cemconres.2004.05.027.
- 497[2]J. Silfwerbrand, Shear bond strength in repaired concrete structures, Mater. Struct. 36498(2003) 419–424. doi:10.1007/bf02481068.
- 499 [3] J. Silfwerbrand, Improving bonding in repaired bridge decks, Concr. Int. (1990) 61–66.
- J. Silfwerbrand, Bonded concrete overlays Research eeds, in: 2nd Int. Symp. Adv.
 Concr. through Sci. Eng., Qubec City (Canada), 2016: pp. 193–205.
- J. Silfwerbrand, H. Beuhausen, Bonded concrete overlays-bond strength issues, in: Int.
 Conf. Concr. Repair, Rehabil. Retrofit., Cape Town, 2005: pp. 19–21.

- J. Silfwerbrand, J. Paulsson, Better Bonding of Bridge Deck Overlays, Concr. Int. October
 (1998) 56–61.
- 506 [7] B.A. Tayeh, B.H. Abu Bakar, M.A. Megat Johari, Characterization of the interfacial bond
 507 between old concrete substrate and ultra high performance fiber concrete repair
 508 composite, Mater. Struct. 46 (2012) 743–753. doi:10.1617/s11527-012-9931-1.
- L. Courard, J.F. Lenaers, F. Michel, A. Garbacz, Saturation level of the superficial zone of concrete and adhesion of repair systems, Constr. Build. Mater. 25 (2011) 2488–2494.
 doi:10.1016/j.conbuildmat.2010.11.076.
- 512 [9] J.S. Wall, N.G. Shrive, Factors Affecting Bond Between New and Old Concrete, Mater. J.
 513 85 (1988) 117–125.
- [10] A.J. Shearrer, K.A. Riding, R.J. Peterman, Effects of Concrete Moisture on Polymer
 Overlay Bond Over New Concrete, K-TRAN: KSU-13-3. (2015).
- 516 [11] H. Beushausen, The Influence of Precast Surface Moisture Condition on Overlay Bond
 517 Strength, Concr. Plant Int. 1 (2015) 144–147.
- 518 [12] H. Beushausen, B. Höhlig, M. Talotti, The influence of substrate moisture preparation on
 519 bond strength of concrete overlays and the microstructure of the OTZ, Cem. Concr. Res.
 520 92 (2017) 84–91. doi:10.1016/j.cemconres.2016.11.017.
- [13] M. Lukovic, G. Ye, Effect of moisture exchange on interface formation in the repair
 system studied by X-ray absorption, Materials (Basel). 9 (2016). doi:10.3390/ma9010002.
- 523 [14] D.P. Bentz, 30 Years of Imaging and Modeling Building Materials at NBS/NIST: From
 524 PIXAR to MICROCHAR, in: Imaging Constr. Mater. Geomaterials, Paris, France, 2016.
- [15] B. Bissonnette, A. Vaysburd, K. Von Fay, Best Practices for Preparing Concrete Surfaces
 Prior to Repairs and Overlays, Denver, CO, 2012.
- [16] Z. Haber, I. De la Varga, B.A. Graybeal, Performance of grouted Connections for
 prefabricated Bridge Elements. Part II: Component-Level Investigation on bond and
 Cracking, in: 2016 PCI Conv. Natl. Bridg. Conf., Nashville, TN, 2016.
- 530 [17] M. Beyene, J.F. Munoz, R. Meininger, C. Di Bella, Effect of Internal Curing as Mitigation
 531 to Minimize ASR Damage, ACI Mater. J. (n.d.) (submitted).
- 532 [18] S. Diamond, J. Huang, The ITZ in concrete a diff€erent view based on image analysis
 533 and SEM observations, Cem. Concr. Compos. 23 (2001) 179–188.
- A. Elsharief, M.D. Cohen, J. Olek, Influence of aggregate size, water cement ratio and age
 on the microstructure of the interfacial transition zone, Cem. Concr. Res. 33 (2003) 1837–
 1849. doi:10.1016/S0008-8846(03)00205-9.
- J.B. Pawley, Fundamental Limits in Confocal Microscopy, in: Handb. Biol. Confocal
 Microsc., Springer US, Boston, MA, 2006: pp. 20–42. doi:10.1007/978-0-387-45524-2_2.
- J.P. Ollivier, J.C. Maso, B. Bourdette, Interfacial transition zone in concrete, Adv. Cem.
 Based Mater. 2 (1995) 30–38. doi:10.1016/1065-7355(95)90037-3.
- 541 [22] K.L. Scrivener, A.K. Crumbie, P. Laugesen, The Interfacial Transition Zone (ITZ)
 542 Between Cement Paste and Aggregate in Concrete, Interface Sci. 12 (2004) 411–421.
 543 doi:10.1023/B:INTS.0000042339.92990.4c.

- 544 [23] E.J. Garboczi, D.P. Bentz, Digital simulation of the aggregate-cement paste interfacial
 545 zone in concrete, J. Mater. Res. 6 (1991) 196–201. doi:10.1557/JMR.1991.0196.
- 546 [24] I. De la Varga, Z. Haber, B.A. Graybeal, Bond of Field-Cast Grouts to Precast Concrete
 547 Elements. FHWA-HRT-16-081, McLean, VA, 2017.