# TRANSPORTATION RESEARCH BOARD NATIONAL RESEARCH COUNCIL



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## Report of Investigation

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#### NEW CORROSION RESISTANT LOW CARBON STEELS FOR CONCRETE REINFORCEMENT

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### NEW CORROSION-RESISTANT LOW-CARBON STEELS FOR CONCRETE REINFORCEMENT

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#### **EXECUTIVE SUMMARY**

Corrosion of reinforcing steel in reinforced concrete structures is placing a significant economic burden on public agencies and private owners. Work investigating the use and affect of improved concrete materials, modernized mixing processes, and refined placement processes has been extensively studied and documented. The benefits from the research are dependent only on the weak link in the production, processing, and placement of the concrete. Quality materials followed by inadequate processing or placement leads to damaged structures.

A complimentary approach to improving the corrosion resistance of reinforcement in concrete structures is to microstructurally design the steel to provide required strengths and improved electrochemical characteristics. The objective of this project was to investigate the effects of the production process and resulting microstructure on the mechanical and electrochemical characteristics of steel embedded in concrete. Dual-phase ferritic martensitic (DFM) reinforcing steel was produced "on-line" for comparison with ASTM A615 and A706 reinforcing steels.

Microstructural analysis and stress-strain testing of intercritically quenched DFM steel with various lath martensite volumes determined that the yield strength is a polynomial function of the lath martensite volume. DFM steel with approximately 30% lath martensite will produce a steel with a yield strength similar to or superior to ASTM A615 and ASTM A706 reinforcing steels. Total elongation values for DFM steels with less than 50% lath martensite exceeded the total elongation values for both ASTM A615 and A706 reinforcing steels. Hence, DFM steels can be economically produced to provide adequate strength and ductility for reinforcement in concrete structures.

Electrochemical testing was carried out under two conditions; in concrete exposed to accelerated corrosive conditions and in electrochemical cells exposed to decanted cement-chloride solution. Testing in concrete followed the Southern Exposure (SE) test method.

Results from the SE testing indicate that DFM steel is more resistant than ASTM grade 60, A615 and A706 reinforcing steels when exposed to wetting and drying cycles with 3.5 wt.% sodium chloride solution. Results from the imposed polarization testing found that the DFM steel is more resistant than ASTM grade 60, A615 and A706 reinforcing steels when immersed in a 3.5% sodium chloride-decanted cement solution and anodically polarized 50mV from the free potential. These results indicate that the production process and resulting microstructure have an affect on the electrochemical characteristics and correctly designing the microstructure can improve the corrosion resistance when exposed to chloride ions. ASTM G61 results did not correlate well with the Southern Exposure mass loss or imposed polarization results.

#### **IDEA PRODUCT**

A dual-phase ferritic martensitic reinforcing steel has been designed and produced to provide adequate strength levels, improved ductility, and improved resistance to chloride induced corrosion when embedded in concrete. Alloying effects and production methods have been investigated to optimize production processes in order to provide a high quality, economically feasible reinforcing steel for use in aggressive environments.

### **CONCEPT AND INNOVATION**

Dual-phase Ferritic Martensitic (DFM) steel is a low alloy, low carbon steel which is produced by simply quenching the alloy from the two phase ferrite/austenite field, thus producing a mixture of ferrite and martensite. In order to identify benefits or potential barriers associated with producing DFM in standard steel mills, it is essential that a brief overview of steel making procedures be provided.

The modern practice for producing reinforcing steel is to utilize mini-mills, which generally have capacities of up to one-million tons per year per plant. In this, steel scrap is melted to a given % carbon (0.3%-0.8%), continuously cast into billets and hot rolled. It is usually normalized (air-cooled) from the finish rolling temperature so as to give a pearlite microstructure. This pearlitic structure from a metallurgical viewpoint is far from ideal and can result in detrimental steel characteristics. The advantages of this process is that the steels satisfy localized construction needs with low cost products, in part due to lower transportation costs and unsophisticated processing.

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The general function of steel bar mills is twofold; first, to convert the melt into a continuously cast, rollable product, and secondly to roll the steel into the desired cross-section. The production process for bars usually begins with melting of the scrap or ingot an pouring into cup-shaped ladles after the melt is sufficiently fluid. Any alloying elements required for the final composition are then added to the ladle. With most bar mills, the molten metal is cast and rolled into long slab-lengths with square or slightly oblong cross-sections in a blooming mill. These slabs are often referred to as blooms and generally range in cross-sections from 150 mm x 150 mm (6 inch x6 inch) to 250 mm x 300 mm (10 inch x12 inch).

After rolling the blooms, it is common practice to cool and store the blooms as opposed to direct continuous rolling of finished products. This practice is primarily economic, since most mills produce many different sizes, shapes, and quality products which are dependent on the final rolling schedule.

The microstructure is very dependent on final composition of the steel and the finish rolling schedules and temperatures of the steel product. The general process consist of heating the bloom and decreasing the cross-section by successively passing the product through smaller and smaller rollers until the final shape is achieved. It should be noted that the steel is more plastic at higher temperatures and requires less work to roll at these temperatures. Unfortunately, rolling at higher temperatures often results in larger grain sizes, which can be deleterious to the mechanical properties. The rolling temperatures are dependent on the equipment available, desired microstructure, composition, and size of the finished product. One objective of the current research is to develop a plan to produce DFM bars in existing mills with minimal modifications.

DFM bars can be designed such that commonly used steel mill finish rolling temperatures can result in the desired two-phase ferritic martensitic microstructure. These temperatures are determined namely by the roll pressures which increase as the temperature is lowered. Thus in "old mills", it is difficult to finish roll below 1000°C due to the inability of the mill to roll at higher pressures. To facilitate rolling at these higher temperatures during the production of DFM, alloying elements can be added to shift the  $\alpha + \gamma$  field, thus making older mills acceptable for the production of DFM bars.

Even though several reasonably simple and inexpensive processes are available, most mills do not have quenching facilities. But, due to market demands and competition in the steel industry, the tide may be changing. Currently, two mills in Oregon and Florida have been equipped with quenching facilities to optimize the microstructure and performance of steel products [Darwin, 1994]. Current negotiations are underway to implement quenching processes and equipment for a third mill in the U.S.

As with conventional reinforcement, the characteristics of DFM steels are very dependent on manufacturing processes. Although DFM steels can be produced with several different thermal and thermomechanical processes, the objective is to utilize common existing production methods to produce DFM microstructure. The simplicity in producing DFM is based on the initial design using the phase diagram (Figure 1a). The weight % carbon and the finish rolling temperature (i.e. the holding temperature in the  $\alpha/\gamma$  phase field from which the steel is quenched) determines the volume fraction of the ferrite and martensite phases. Because the mechanical properties and electro-chemical characteristics of the steel are very dependent on the microstructure, it is essential that production schemes are well planned. In part, this can be accomplished using the lever rule which determines the percent quantity and carbon content of each phase. This rule is given by the intercept of the tie-line at a given temperature and shows the fractions of phases and compositions in equilibrium at that temperature (e.g. Figure 1). In addition to the final rolling temperature, rolling schemes which minimize grain size must be developed. It should be noted that in order to cost effectively produce DFM steel, a scheme which utilizes existing production methods must be utilized to produce the most beneficial microstructure.



Figure 1a





The mechanical properties of DFM steel are determined by the composite morphology, the martensite volume, the grain sizes, the carbon content, and the type and amount of alloying elements. The final

morphology, martensite volume, grain sizes, and carbon content of the phases are dependent on the production method. Alloying elements can be used to optimize these characteristics. Figure 1b is an example of how finish rolling temperatures can be manipulated with alloying elements. The addition of 2% silicon decreases the slope of the  $A_3$  line and increases the overall finish temperatures. Because existing mills tend to roll at higher temperatures, such additions of silicon are beneficial for the production of DFM steel. Note also that because the slope of the  $A_3$  line decreases, deviations in the finish rolling temperature will have less effect on the percent volume and carbon content of the different phases. This indicates that temperature control is not a limiting factor.

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The important design aspect for corrosion resistance is to prevent formation of carbides at the ferritemartensite interface. This can be avoided by properly designing the composition and rolling and quenching schedule. Figure 2 shows some typical heat treatment processes used to produce different DFM microstructures. These microstructures result in different characteristics for DFM bars and the final required microstructure will depend on the application of the product and the available manufacturing process. As described earlier, if quenching facilities were available at the end of the processing line, most rolling mills would produce the dual-phase microstructure with elongated grains using the thermo-mechanical process.



FIGURE 2 Different production processes for Fermar steel giving different morphologies

From our earlier research on low carbon and high strength low alloy (HSLA) steels, it was established that superior mechanical properties (especially ductility and toughness) of martensitic steels could only be achieved if the martensite is of the "dislocated lath" type rather than the "twinned plate" type. This difference in morphology is related to the composition and austenite to martensite transformation temperature. In general, the carbon content must be below approximately 0.35 wt. % and the martensite temperature above approximately 350 °C in order to avoid twinned plate martensite. Thus, in producing DFM steels, the tie line (hence, the finish temperature) at the desired volume fraction of austenite (to produce lath martensite) must comply with the above conditions. For the DFM steels developed here with a 0.1% carbon content, the lowest temperature will be approximately 800°C (Figure 1b) for finish control rolling prior to water quenching.

Controlled rolled DFM steels were produced both in our steel production laboratory and commercially produced by controlled rolling and water quenching. Both productions resulted in fine grained, carbide free, dual-phase ferritic martensitic microstructures. Figure 3 shows an example of the lath martensite structure in the DFM steels tested in this project. Results to date indicate that the dual-phase microstructure can provide adequate strength and ductility requirements for reinforcing bar specifications and is more resistant than ASTM grade 60, A615 and A706 reinforcing steels when embedded in concrete.



FIGURE 3 TEM micrograph showing lath martensite

### **IDEA PROJECT INVESTIGATIONS AND PROGRESS**

The DFM research project consisted of three general stages. Stage 1 of the project included a literature review of high performance reinforcement steels and consisted of planning heat treatment schemes for the production of the DFM reinforcing bar. Stage 2 investigated the mechanical properties/microstructure relationships for different manufacturing processes. Stage 3 investigated the electrochemical microstructure relationships for the microstructure which provided adequate properties from the stage 2 mechanical testing.

#### STEEL PROCESSING

A heat of the chemical composition shown in Table 1 was melted for the production of #5 reinforcing bars. Small samples were control rolled and quenched from the dual-phase region to produce DFM microstructures with varying quantities of lath martensite. Finish control rolling was carried out at 890°C, 910°C, 930°C, 950°C, 960°C, 1000°C, and 1050°C respectively. Following these treatments, the samples were immediately quenched in a 3% brine solution. Three separate samples at each temperature were produced.

С	Mn	Si	S	Р	Cr	Ni	Mo	Cu	Sn	Al	N	Fe
											(ppm)	
0.105	0.49	1.81	0.018	0.019	0.11	0.13	0.025	0.37	0.021	0.008	95	Re

#### TABLE 1 DFM Chemical Composition (wt.%)

The samples were then ground with SiO sand papers and polished down to  $1\mu m$  with diamond paste. All samples were etched with a 5% nital etch and observed in the optical microscope to determine martensite volumes. For each sample, three volume fraction analyses were performed: three at the outer edge of the bar, three at the quarter point of the cross-section, and three at the center of the sample. Using computer software and quantitative computing programs, the volume percentages of the "dark-etched" and "light-etched" phases were determined. The averaged results of these measurements are shown in Table 2. The light-etched phase was identified to be ferrite. Using the Transmission Electron Microscope (TEM), the dark-etched phase was proved to be lath martensite. A TEM micrograph of the lath structure is shown in Figure 3. No interface carbides were observed. This phase of the project determined that the steel should be control rolled at 850°C, followed by an immediate water quench. This heat treating process produced #5 DFM reinforcing steel with 25-30% martensite. Grain sizes for all sample ranged from  $3\mu m$  to  $6\mu m$ . Approximately 600 kg of #5 bars were then control rolled and quenched for further testing. Optical micrograph investigations of this second batch determined that the steel microstructure consisted of approximately 25% lath martensite in a field of equiaxed ferrite (herein referred to as AR-DFM samples). No interface carbides were observed. These samples were used for all testing.

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		Theoretical Data			
Heat Temp. (°C)	% Ms at Center	%Ms at 1/4 pt.	% Ms at edge	Avg. % Ms	% Ms
890	30	36	41	36	28
910	35	45	48	43	35
930	31	36	45	37	34
950	36	37	44	39	35
<b>96</b> 0	38	47	62	49	37
1000	41	49	57	49	42
1050	40	56	76	57	50

TABLE :	2	Experimental	and	theoretical	l data	of r	nartensite	volumes
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#### **MECHANICAL TESTING**

Stage 2 of the project included characterizing the mechanical properties of the DFM steel specimens. In order to determine the effect of the martensite volume on the yield strength, tensile strength, and total elongation, AR-DFM samples were heat treated at three different temperatures. Four samples each were heated to 800°C, 950°C, and 1100°C and cold water quenched so as to obtain approximately 20, 50, and 80 percent martensite respectively. Following the heat treatments, optical microscopy revealed that the actual average martensite volumes were 20% for the sample treated at 800°C, 47% for the samples treated at 950°C, and 75% for the samples treated at 1100°C. The samples were then fabricated as shown in Figure 4 for monotonic stress-strain testing. Monotonic stress strain testing was carried out on a 120 kip Satec/Baldwin testing machine following ASTM E8 standards under strain controlled conditions. The testing strain rate was 0.003 in./in/min. All stress strain data was recorded and stored utilizing an IBM compatible computer equipped with data acquisition software. Tensile strengths and elongation values were obtained from the stress-strain data and results are shown in Figure 5. Table 3 shows yield strengths, ultimate strengths, and total elongation values for each of the heat treated AR-DFM samples.



FIGURE 4 Stress strain sample



FIGURE 5 Stress strain results of DRM steel with varying Ms volumes compared with grade 60 A615 and A706 steels

TABLE 3         Mechanica	l characteristics of DRM	steels with (	different martensite	volumes
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	Strength,	, MPa (ksi)	Total
Sample	Yield	Ultimate	Elongation (%)
AR20	455 (66)	779 (113)	20
AR47	510 (74)	814 (118)	13.5
AR75	670 (97)	848 (123)	6

#### **ELECTROCHEMICAL TESTING**

To evaluate the corrosion characteristics of the reinforcing steels, electrochemical testing was completed. Three test procedures were carried out to evaluate the different steels. The first set of testing, ASTM G-61, *Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Base Alloys,* was carried out to evaluate the susceptibility of the different steels to localized corrosion. The second set of testing, the Southern Exposure test method, consisted of measuring and evaluating macrocell current between different reinforcing mats when the reinforcing bars are embedded in concrete. The third and final set of electrochemical testing was carried out to investigate the susceptibility of the different microstructures on the corrosion activities of the different steels. This testing has determined the corrosion susceptibility of the different steels when exposed to chlorides and has provided information on microstructural effects on corrosion activity.  $(\cdot, \cdot)$ 

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#### **Polarization Testing**

Stage 3 of the investigation consisted of determining the electrochemical characteristics of the grade 60 A615, A706, and DFM reinforcing steels subjected to simulated and actual concrete environments. To initially characterize the steels, ASTM G-61, *Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Base Alloys*, was performed on each type of steel in two different environments.

The first environment consisted of a decanted cement solution. The solution was made by mixing 2 parts water with 1 part Type I/II cement for 30 minutes and then allowing the cement to settle for 1 hour. The solution was then decanted and stored in polyethylene containers for testing. Table 4 shows the chemical composition of the solution after 2 and 16 weeks of mixing determined with atomic emission spectroscopy analyses. The second test solution consisted of a 3.56% sodium chloride/decanted cement solution. The initial proposal recommended adding Welam gum to increase the viscosity of the solution and prevent mixing of the solution at the steel/solution interface during testing. This was recommended to better simulate actual concrete environments. Because the gum is extremely hydrophobic, testing incorporating this product was not feasible.

The scan rate for the anodic polarization (ASTM G-61) testing was 1 mV/sec. Table 5 shows the corrosion potential, the potential where localized corrosion initiated, and the difference in these potentials. These results tend to indicate that the DFM steel is more susceptible to chloride induced corrosion in a deaerated decanted cement solution. Figure 6 shows the anodic polarization plots.

Element	μg/l (2 weeks)	μg/l (16 weeks)
Al	306	3683
Ba	446	612.5
Ca	1360000	1302500
Cr	2580	2792.5
Fe	11	3398
K	1240000	1235000
Li	177	165.75
Mg	44	1786.5
Mn	3.3	32.775
Мо	420	466.25
Na	, 221000	210500
Sr	9080	8850
Zn	141	88.9
pH	12.8	12.8

#### TABLE 4 Atomic emission spectroscopy analyses of decanted cement solution

 TABLE 5 Corrosion potential and localized corrosion potential values

Sample	$\Delta O_{\rm corr}$	$\Delta O$ <sub>localized corrosion</sub>	$\Delta(\Delta O)$
ASTM A-615	-0.459 V	0.260	0.719
ASTM A-706	-0.457	0.338	0.795
DFM	-0.622	-0.298	0.385



FIGURE 6 ASTM G-61 results for ASTM A706, ASTM A615 and DFM reinforcing steels

#### **Southern Exposure Testing**

The modified Southern Exposure test method is believed to simulate 15-20 years of marine exposure or 30-40 years of deicing treated bridge exposure. The schematic of the Southern Exposure test sample is shown in Figure 7. By electrically connecting the upper and lower mats and subjecting these mats to different environmental conditions (e.g. chloride content), potential differences are set up between the mats and corrosion is induced.

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FIGURE 7 Southern Exposure schematic

A total of thirty samples (10 with DFM steel, 10 with ASTM grade 60 A615, and 10 with ASTM grade 60 A706 steel) were cast. The water cement ratio was 0.68. Table 6 shows the material descriptions and mix design. All samples were wet cured for three days followed by 25 days of air curing. Average compressive strength for the concrete samples cured as described above was 20 MPa (2800 psi). Chloride permeability testing and evaluation followed AASHTO T-277 - *Rapid Determination of the Chloride Permeability of Concrete*. Testing was carried out at 28 days and results indicated that all samples were highly permeable.

TABLE 6	Mix design	and description	on of materials	s used to	make SE s	pecimens
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Material	Description	Mass, kg/m <sup>3</sup> (#/cy)
Coarse Aggregate	9 mm (3/8") MSA river gravel	3441 (1196)
Fine Aggregate	F.M. = 3.09, BSG = 2.67	4785 (1663)
Water	Tap Water	1108 (385)
Cement	Type I/II	1630 (566)

Macrocell current values were recorded daily beginning 28 days after casting. Alternate wetting and drying cycles were such that the samples were ponded seven days followed by seven days of drying. Figure 8 shows results from the testing. Following 1 year exposure, steel reinforcing samples were removed from the concrete and ASTM G1 mass loss evaluations were performed on the top bars of 5 samples from each steel

type. Figure 9 shows these results. It can be seen that the southern exposure results are not as indicative of corrosion damage as are the mass loss results. It is believed that because chloride induced corrosion is mostly associated with localized microcell corrosion, macrocell results may not be representative of the degree of corrosion induced damage caused by chlorides.



FIGURE 8 Southern Exposure test results



FIGURE 9 ASTM G1 Mass loss results

#### **Imposed Polarization in Deaerated Decanted Cement Solution**

Testing of the three different steel types in simulated concrete environments was carried out to determine the effect of microstructure on the corrosion characteristics. Decanted cement solution was utilized as the electrolyte.

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Steel samples were cut to approximately 25 mm lengths (1 inch) and then polished to 1 micron. Three millimeter (1/8 inch) copper wires approximately 200 mm (8 inches) in length were attached to the side opposite the polished surface. The entire sample except approximately 100 square millimeters on the polished surface was coated with a glyptal paint. After curing, the samples were placed in cells as shown in Figure 10. Each cell initially contained 1 liter of decanted cement solution. High purity nitrogen was bubbled throughout the cells for the entire test period. After quasi-steady state conditions were achieved, a solution containing 40 grams NaCl and 100 grams decanted cement solution was added to each cell to make the cell solution approximately 3.5 wt.% NaCl. Each sample was then polarized 50 mV anodic of the free potential. Current density values were recorded every 15 minutes for 21 days using a data acquisition program. Results from the tests are shown in Figure 11. Figure 12 shows representative samples from both the DFM and A615 bars. The excessive corrosion of the A615 bar compared to the apparent non attack of DFM is obvious.



FIGURE 10 Corrosion cell layout



FIGURE 11 Imposed polarization test results



FIGURE 12 Specimens after anodic polarization in deaerated 3.5 wt.% NaCl/decanted cement solution

#### PLANS FOR IMPLEMENTATION

The University of California at Berkeley joint research team in the Department of Mineral Engineering and Materials Science and the Department of Civil and Environmental Engineering has begun collaboration with a major bar producing US steel company to produce commercial grade DFM reinforcing bars. A program has been agreed upon and the project will commence upon funding of the second year.

The steel company will melt, cast, and roll into 16 mm reinforcing bars a 50 ton heat of low carbon steel as defined by our year 1 research (approximately Fe/2Si/0.8C). The company will arrange for the bars to be heat treated to specifications so as to obtain the requisite microstructure and mechanical properties. The approximate investment by the steel company will be approximately \$50,000. The partnering plant does not

have water quenching facilities and will have to heat treat the steels after rolling. It is believed that successful results will spur the steel producers to invest in on-line quenching, for which reinforcing bar production will be profitable.

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The research team at the University will require follow-up funding to oversee and optimize production process, characterize the resulting microstructures, and confirm mechanical and electrochemical properties.

The research investigating the mechanical and electrochemical characteristics of DFM steel is now ready to commence year 2 of the research in order to implement commercial production of DFM steel reinforcing bars.

#### CONCLUSION

The mechanical properties of DFM steel can be varied depending on the final roll temperature. Results from this study indicate that grade 60 DFM steel reinforcing bars can be commercially produced with a Fe/2Si/0.8C steel. Finish rolling temperature will be approximately 850°C followed by an immediate water quench. This heat treating process will produce a DFM reinforcing steel with 25-30% lath martensite, yield strengths between 480 MPa (67 ksi) to 515 MPa (72 ksi), ultimate tensile strengths of approximately 850 MPa (120 ksi), and total elongation of approximately 18%.

Electrochemical testing indicates that DFM steel is more resistant than conventional A615 and A706 grade 60 reinforcing steels when embedded in concrete and subjected to an aggressive chloride environment. ASTM G61 results did not correlate with the long term test results.

#### **INVESTIGATOR PROFILE**

This study was directed by Gareth Thomas, Professor in the Material Science and Mineral Engineering Department at the University of California at Berkeley and Ben Gerwick, Professor Emeritus in the Department of Civil and Environmental Engineering Department. Professor Thomas has been elected to both the National Academy of Sciences and the Academy of Engineering. His main interests are in understanding the effects of microstructure, morphology, and local composition upon the properties of materials and utilizing this knowledge to economically design improved or new materials. He has authored or co-authored over 550 publications, including three books.

Ben Gerwick is Chairman of the Board of Ben C. Gerwick, Inc. and, in addition to his many honors and awards, is a member the National Academy of Engineering, Past-Chairman of the Marine Board of the National Research Council, and Past-Director of the American Concrete Institute. His interests include design, construction, and performance of offshore concrete materials and structures. He has authored or coauthored over 225 publications, including nine books.

Laboratories in the Civil and Environmental Engineering (CEE) Department and the Material Science and Mineral Engineering (MSME) Department were utilized for this research program. The CEE department utilized equipment to mix and cure the reinforced concrete specimens. In addition, temperature and humidity controlled rooms in the CEE labs were utilized to subject the specimens to an accelerated corrosive environment. The MSME department provided various types of electron microscopes, x-ray, and other characterization facilities and equipment to characterize the microstructure of the various samples. In addition, laboratories in both the CEE and MSME departments were utilized to investigate the corrosion susceptibility of DFM steel in simulated and actual concrete environments. Heat treating facilities, machine shops, and carpenter shops were utilized from both laboratories to produce and fabricate steel testing specimens and concrete formwork.

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