The Invention of Ductile Iron...In Millis' Own Words

The late Keith Millis recounted his discovery during a 1983 Ductile Iron Society speech celebrating the 40th anniversary of his laboratory invention.

Keith D. Millis
Ductile Iron Society

"Early in the second World War, INCO (International Nickel Co.) was promoting Ni-Hard, a very hard cast iron used in abrasion-resistant applications such as ore grinding balls and crusher rolls. In addition to nickel (Ni), the iron required chromium (Cr) to the tune of 1.5% to assure that all the carbon (C) was combined as a hard carbide. Cr was considered critical to the war effort and the supply was not that great. The development side of R&D at INCO panicked with fear that the supply of Cr would be depleted and not available for Ni-Hard. A development engineer, Kenneth A. DeLonge, was responsible for promoting the Ni-Hard development in the field. On January 9, 1942, he wrote a memo to the laboratory outlining their concern and requesting that the laboratory look for a substitute for Cr. The head of the laboratory, Mr. Norman B. Pilling, accepted the job and it was assigned to me to do the research.

Gagnebin (l) and Millis congratulate each other upon receiving AFS Gold Medals in 1952 for their invention.

In the last 25 years, I have complained on occasion that researchers, in the vernacular, reinvent the wheel. They do not research the literature, and very often we see reports of expensive research that duplicates results already in the literature. I was quite new in 1942 in the cast iron section, having just transferred after a year or so in the nonferrous section. My total literature research involved only consulting a book on chemical compounds to find out which elements combined with C to form carbides…I had written down a list of elements that I thought might form carbides…and magnesium (Mg) showed two carbides, Mg2C3 and MgC2.

I then laid out a program in which the following additions should be made: Cr, zirconium (Zr), cerium (Ce), bismuth (Bi), copper (Cu) and lead (Pb), tellerium (Te) - two levels since it was known to form carbides in iron - Mg and columbium (Cb). I went over the plans with my superiors and was immediately told that I could make all the additions except Mg. That was forbidden because it was dangerous. As I recall, during Mr. Pilling's actual research days, he was experimenting with Mg additions in high-Ni iron alloys. Mg is commonly used in Ni alloys such as Monel for deoxidization purposes. He found that where the iron (Fe) content approached 25% or more, the violence of the Mg addition became intolerable. Hence, he forbade me to use Mg.

This event showed up later on in the litigation with Ford, as another example of 'I don't get no respect.' In his testimony, Pilling said, 'When Millis had prepared his program which had involved making experimental melts of nickel-irons containing a variety of elements, he listed what he thought might have interest as carbide stabilizers. When I saw the list and found on it Mg, I was first disposed to tell him to scratch it off and forget it. But perhaps a little charitable thought occurred. It seemed to me, well, we all have to learn, sometimes the hard way. Go ahead and do it.'

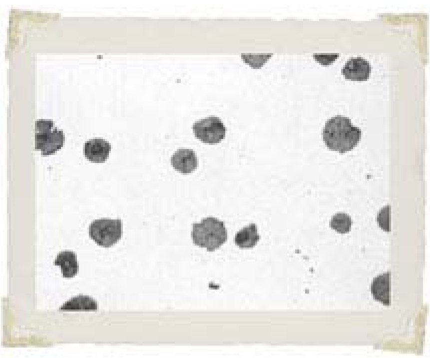
The Master Judge Rifkind broke in and said: 'He was expendable?' To which Mr. Pilling replied: 'He was expendable, and I was not.'

The fact that Mg could form two carbides with C was no guarantee that it would have this action in a molten cast iron bath. Here, I might refute my own earlier statement about researchers doing too little in the way of checking out past work in the area. For had I searched, about all I would have found about Mg in cast iron would have been the Meehan patent which put Mg in the same category with calcium (Ca) as an element that promoted the breakdown of carbide and the precipitation of graphite as flakes. With this knowledge, I probably would have scratched Mg myself.

"Max Kuniansky had just hired me before the 1948 convention. All of you who knew him know how impetuous he was. He jumped on ductile iron like a duck on a junie-bug. After the announcement, he rushed out and called the scrap dealer in Lynchburg and had a truckload of Mg aircraft engines delivered to the foundry. Fortunately, I paid a little more attention to the paper, so I knew this wasn't the way you did it."

- Harvey Henderson, retired, Lynchburg Foundry

Well, after I got clearance from above, and was properly warned about the incompatibility of Mg and molten iron, I planned my heat to test the elements that seemed to have the most promise. It was a 250-lb melt produced in the Detroit Rocking Furnace on February 13, 1942. It was a normal base Ni-Hard composition, 3.35% C, 0.50% Si, 0.50% Mn, 4.5% Ni, but without Cr. The elements I investigated were Zr, Ce, Bi, Cu plus Pb, two levels of Te, Cb and 0.5% Mg. The other taps were the base iron and one with 1.50% Cr - normal Ni-Hard. Each tap was cast into a 2 x 6 x 6-in. chill block, chilled against a 2 x 6-in. face.



The microsamples of the heats tapped on April 12, 1943 showed that the graphite had taken on a spheroidal shape…and ductile iron was born.

The chill blocks were subsequently fractured. The base iron had 0.02 in. of carbidic iron above the chill face and all except the bottom 0.33 in. was graphitic. Of course, the Cr-containing iron was completely carbidic - which was the goal. None of the others showed significant carbides except the Te, which was not enough and Mg - which was really a surprise. It had 5 in. (out of 6) that was carbidic. And even the top inch included some carbide. Also, the block was considerably tougher, more difficult to fracture than the regular Ni-Hard block.

This result, plus the fact that Mg was most-effective in lowering the sulfur (S) content of the iron was considered novel, which is a criterion for patent approval. Accordingly, the result was recorded on February 14, 1942, witnessed by A.P. Gagnebin. A patent suggestion was written and eventually I was issued patent number 2,516,524 on July 25, 1956.

In January 1946, out of curiosity, I got the chill block out of storage and polished a section of the inch at the top. If I had done this back in 1942, we could have celebrated our 40th anniversary last year because the graphite in the top was spheroidal. How many discoveries do you suppose go undiscovered permanently under similar circumstances? This research was followed by another program of the effect of Mg on white iron having no Ni content. Nothing new was learned and the program on white iron was closed out. After discussions about Mg in iron and the fact that Mg had a very pronounced effect on cast iron, it was natural to wonder if it would be effective in any way in a cast iron of a composition and inoculation procedure designed to produce graphite in the iron. On March 17, 1943, N.B. Pilling, who was head of the lab, made it official in a memo directing that a research program be undertaken to determine the effect of Mg in gray iron. This project was assigned to A.P. Gagnebin and me. There had been some early evidence that Mg would improve a gray iron. Early in January of 1943, several taps of a heat in the white iron investigation were treated with Mg and inoculated with 0.5% Si as Fe85%Si. The transverse

What it All Meant…

The results of Millis' and Gagnebin's "taming" of the reaction between Mg and cast iron would have a huge impact on the industrial world. Millis found that the tensile strength of high-carbon gray iron had increased from the expected 18,000 lb/sq in. level in the base iron to an unexpected 112,000 lb/sq in. level with the iron treated with Mg. An immediate examination under the microscope showed that the graphite was present in spheroidal form - a structure never before seen in as-cast gray iron.

In traditional gray iron, the graphite flakes act as stress raisers that can initiate the propagation of cracks. In ductile iron, a suitable treatment of the molten iron causes the graphite to precipitate as spheroids rather than flakes. The nearly spherical shape of the graphite removes the "crack" effect and, in fact, the graphite spheroids act as "crack arresters."

The result of Millis' experiments was a new cast iron several times stronger than gray iron, 4-12 times tougher and so ductile that it could be bent and twisted without breaking. This new ductile iron family, to a large degree, bridged the gap between gray iron and steel. It created the processing advantages of gray iron - low melting point, good fluidity and castability and ready machinability - combined with many of the engineering advantages of steel, including high strength, toughness, ductility and wear resistance. It would permit the production of castings that are intricately shaped or have very light sections that could still withstand severe service conditions.

Since Millis' discovery in 1943, a whole host of goods found a new mode of production, and more than 88 million tons of ductile iron castings have been produced in the U.S. alone.▶

Ductile iron machined flat from a casting and then twisted.

strength of the iron increased from about 4500 lb to 6500 lb. The graphite was finer in the treated iron, but still flake.

Under the new job, two heats were made on April 12, 1943. One was a base iron of 3.64%C, 2% Si, 0.75%Mn, 0.06%S and 2%Ni. The other iron was a higher strength, lower carbon equivalent (CE) base iron and while the results were similar, they were not as startling as with the high CE heat. Mg additions, as an 80% Ni-20% Mg alloy of 0.05%, 0.30%, 0.40% and 0.50% were made, each followed by a FeSi inoculation. Arbitration bars were broken and tensile bars machined from the arbitration bars…. It didn't take long to cut several microsamples, which were polished and examined under the microscope. And of course, they revealed that the graphite was entirely in the spheroidal form.

"Until we had ductile iron, we in the foundry industry were looked upon as the handmaidens of the engineers. We were treated with something that bordered very nearly on contempt. Now, this picture has changed tremendously, and I thank Millis, Gagnebin and Morrogh for proving that we weren't the backwoodsmen of a technological era."

- Ronnie Taylor, Robert Taylor & Co. Iron Foundries, Scotland
(first European licensee)

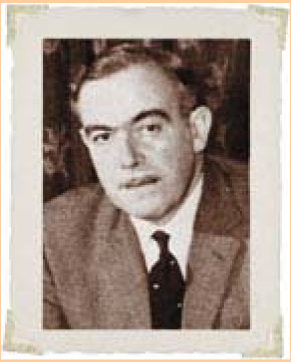
That accounts for the invention. Here was a new material with outstanding properties and economies that from that point on could be produced at will. I, being a very young and naive research metallurgist, thought, 'That's it, let's get the foundry industry going on this.' Of course, at that point, I had had no experience with the foundry industry and its level of technical expertise. There was certainly an awesome amount of work necessary before any information could be released, such as effects of elements, additives, production procedures, physical properties, mechanical properties, corrosion resistance, deleterious elements, heat treatment for various properties and a multitude of other factors that had to be investigated before a patent application could ever be made.

As some insurance against a leak and possible loss of patentability, a page of the basic facts was written in my record book, signed by A.P. Gagnebin and me and witnessed by a notary public. It was dated May 5, 1943.

Because of the potential value of the inoculation, it was decided that if only a few people know about it, the possibility of a leak would be diminished. Consequently, when I was about to make Mg additions to a heat in the experimental foundry, I would make several phone calls to department heads in the main lab. Soon, the foundry phone would ring and people privy to the information would be called into the main lab on some pretext. In the early '50s, I ran into a man who had worked in the lab during most of the period. He expressed wonderment that he could have been there all the time and known nothing about it. This experimentation continued for five years before an application was filed in Washington, D.C. on November 21, 1947.

In order to establish, for the record, on an earlier date an application was filed in Great Britain, March 22, 1947. The U.S. Patent Office really investigated this one, possibly because Henton Morrogh filed January 25, 1949 for a patent on his Ce process in hypereutectic irons. The INCO main patent and the patent on improved cast iron 2,485,760 and 2,485,761, respectively, were granted October 25, 1949. The Ce patent was granted November 15, 1949. Incidentally, INCO subsequently purchased the Ce patents.

Henton Morrogh: Another Leading Man in the Ductile Iron Saga



Henton Morrogh

While Millis and the INCO team's work on ductile iron ended up being the method embraced by industry and the first to be patented, they were not the ones who unveiled the monumental discovery of the new spheroidal material. At the 1948 AFS Casting Congress in Philadelphia, Henton Morrogh of the British Cast Iron Research Assn. (BCIRA) presented "Production of Nodular Graphite Structures in Gray Cast Irons," which documented the work done to produce nodular (ductile) irons by the addition of rare earth elements.

Univ. of Wisconsin's Carl Loper, Jr., recalled in 1973 that Morrogh's paper presentation drew a packed auditorium. "He announced that they were able to produce nodular structures without heat treatment by adding cerium to low-sulfur hypereutectic gray cast iron. In the discussion period that followed immediately thereafter, Mr. Tom Wickenden of INCO came to the podium and announced the results of research underway for some period of time at INCO…Since then, a completely new industry was born."

The June 1948 edition of American Foundryman (the forerunner of modern casting) reported: "H. Morrogh's paper describing the production of cast irons with nodular graphite in the as-cast condition, thus eliminating lengthy heat treatment, was hailed as an important contribution to the fundamental knowledge of metallurgy. Discussors of the paper included many of the foremost gray iron metallurgists of the United States and Canada."

In 1973, Morrogh described the events that unfolded. "After the presentation of my paper, there was this wonderful announcement by Wickenden of this remarkable and revolutionary discovery by Millis and Gagnebin," he recalled. "I had the privilege of learning about the discovery one week earlier and was told they would make the announcement following the paper.

"Almost all of the ductile iron produced commercially has been based on procedures developed by Millis and Gagnebin. This not only required the technical brilliance of their invention, but also the patience and determination of INCO to promote the invention in an ambitious and circumspect manner. Many technical men have represented INCO in these difficult preliminary activities."

While he was the first to announce his discovery, he must certainly have been surprised to see that another party's name was etched in for discovering the landmark metal. But to Morrogh, it appeared that who discovered it was merely incidental. In a 1973 modern casting article, Morrogh wrote: "Twenty-five years ago, I was enthusiastic for the material and its development, and in the ensuing years I have lost none of this enthusiasm. I am convinced that ductile iron will in the future be of increasing importance to the foundryman and engineer alike."

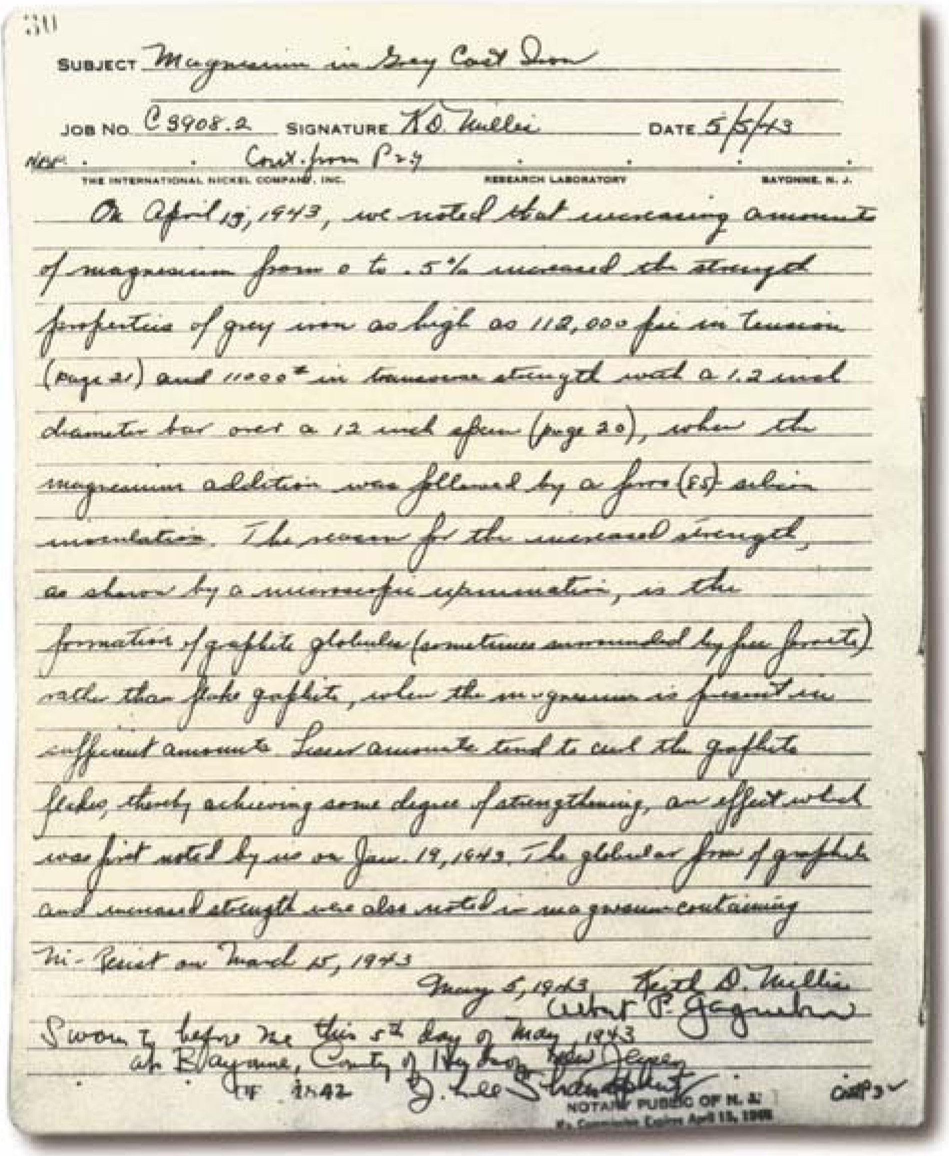
Millis himself was quick to point out Morrogh's important place in the history of ductile iron, and that it was his paper that spurred the invention along. "If Morrogh hadn't delivered that classic paper on cerium on May 7, 1948," said Millis, "who can say when INCO would have made it?"

Reg D. Forrest of Pechiney Electrometallurgie characterized the situation in a 1992 letter. "Imagine the consternation in the INCO laboratories when, just as they were finalizing their plans for announcing and commercializing their development, the news broke that Henton Morrogh of BCIRA was to present a paper at the May, 7 1948, AFS Convention in Philadelphia describing the development of the same material using cerium as the additive," he wrote. 'Somebody, someday should write the book or make the movie of the events leading to the discovery of ductile iron - but they will have difficulty casting the principal characters - Keith Millis and Henton Morrogh."

- Michael J. Lessiter, editor

"After INCO began looking for licensees in 1948, suddenly, after five years of work, we couldn't repeat the spheroids. It meant going through wet chemistry for long list of elements and anxiously waiting for results. We finally found it had to do with a batch of bad pig iron that we received. Keith was under terrific pressure, but just calmly attacked the problem. He may have felt emotion but sure didn't show it."

- Warren Spear, retired, INCO



SUBJECT Magnesium in Grey Cast Iron

JOB NO. C3908.2 SIGNATURE K.D. Millis DATE 5/5/43

Cont. from P 27

THE INTERNATIONAL NICKEL COMPANY, INC. RESEARCH LABORATORY BAYONNE, N. J.

On April 13, 1943, we noted that increasing amounts of magnesium from 0 to .5% increased the strength properties of grey iron as high as 112,000 psi in tension (page 21) and 11000# in transverse strength with a 1.2 inch diameter bar over a 12 inch span (page 20), when the magnesium addition was followed by a ferro (85) silicon inoculation. The reason for the increased strength, as shown by a microscopic examination, is the formation of graphite globules (sometimes surrounded by free ferrite) rather than flake graphite, when the magnesium is present in sufficient amounts. Lesser amounts tend to curl the graphite flakes, thereby achieving some degree of strengthening, an effect which was first noted by us on Jan. 19, 1943. The globular form of graphite and increased strength were also noted in magnesium containing Ni-Resist on March 15, 1943

May 5, 1943 Keith D. Millis

Albert P. Gagnebin

Sworn to before me this 5th day of May, 1943 at Bayonne, County of Hudson, New Jersey

NOTARY PUBLIC OF N. J.

This page in Millis' record book, signed by he and Gagnebin 24 days after the historic heat was poured, secured the tandem's place in history as the co-inventors of ductile iron.

Work continued after the patent filing with full intentions of being able to transfer technology and know-how to licensees. Incidentally, the formation of a license contract and system was a big problem for the legal staff, since INCO had never had a royalty licensing system. We were somewhat distressed to learn that Henton Morrogh was going to reveal his process at the May 7, 1948 AFS meeting in Philadelphia. We didn't feel that we were ready. On the other hand, we knew about his process and knew the Mg process was the better of the two. Consequently, rather than let the American foundry industry 'chase the wrong rabbit,' so to speak, the decision was made to offer a simple announcement that INCO had a process using Mg. This was done as a discussion of Morrogh's classic paper. Then we really went to work both in the lab and at the negotiating table because in effect we were deluged with requests for licenses.

During the years since, there have been many improvements in processing techniques and in the understanding of interactions of elements and primarily the need for close quality control. The Mg reaction in iron has always fascinated me because of its spectacular brilliance. Unfortunately, from this standpoint, improvements in techniques have reduced this spectacular aspect to unimpressive mildness in most shops. OSHA and labor promoted this, but on occasion I will be in a shop and get a glimpse reminiscent of the past.

For the trial with Ford Motor, a noted judge, the honorable Simon H. Rifkind, was appointed as special master to preside over the proceedings. At one point, INCO and Ford moved the hearing to Cooper-Bessemer Corp., at Mount Vernon, Ohio, in order to let Judge Rifkind witness the process and get testimony from three of the Cooper-Bessemer people. After the demonstration, one of the witnesses was asked if he would describe in words what the nature of the reaction is, that is to say, its appearance. Before he could answer, Judge Rifkind broke in saying, 'He has to qualify as a poet for that.' The witness answered, 'Well, at the moment of contact with the hot metal, the Mg-bearing alloy creates a Mg fire which is undoubtedly an oxidation of Mg and a certain percentage of the Mg burned off into the atmosphere, creating a white smoke which is likely Mg oxide.'

At this point, the judge broke in again, saying, 'If you want to find words for it, I will refer you to the early chapters of Exodus, where there is a description of what happened on top of a mountain called Sinai.'

I am sure that everyone in the courtroom hunted up a bible to see what this was. It follows: "And it came to pass on the third day in the morning, that there were thunders and lightnings and a thick cloud upon the mount, and the voice of the trumpet exceeding loud; so that all the people that were in the camp trembled. And Moses brought forth the people out of the camp to meet with God; and they stood at the nether part of the mount. And Mt. Sinai was altogether on a smoke, because the Lord descended upon it in fire; and the smoke thereof ascended as the smoke of a furnace, and the whole mount quaked greatly.' "

"Most people fail to realize the work that Millis and Gagnebin did in their labs. Sure, someone might have come up with the Mg treatment method, but it wouldn't have gained the promotion and stronghold it did with INCO behind it. Those two spent all their time on helping people get started. We all owe them both a big boat of thanks."

- Fred W. Jacobs, retired, Texas Foundries