ANALYTICAL REFERENCE MATERIALS INTERNATIONAL CERTIFICATE OF ANALYSES

Grade: **Invar 36**Part Number (Q.A.NO.): **IARM 24A**

Certification Date: 02/15/1991 Certificate No.: 24A-021591ARM-F

Interpretation of Data

- Certified values listed below reflect analysis results submitted by qualified analytical laboratories using a combination of methods and instrumentation that
 emulate actual methods and instrumental techniques currently utilized in the analytical community.
- 2. Any data reported and enclosed by a **parentheses** () is a "best estimate" and is NOT CERTIFIED. This data could not be quantified sufficiently for certification. It was however, reported by enough laboratories to be considered as potentially present in the matrix of the material being examined.
- 3. The "Interlaboratory Analysis Program" (ILAP) utilized in the establishment of the data are an ongoing program with permanent membership. Certain elements may be selected by a consensus of the members for more extensive testing. Therefore the data in **brackets** [] **indicates further testing is in process.**
- 4. The "Standard Deviation" using an N-1 calculation is also enclosed by a parentheses () below the elements certified concentration.

Important: A "User Registration Card" accompanies all shipments. This card should be completed immediately upon receipt of materials with the appropriate user information. This is the only way in which ARMI can guarantee customer updates or possible data modifications!

| <u>Carbon</u> | Manganese | Phosphorus | <u>Sulfur</u> | <u>Silicon</u> | Chromium |
|-----------------|-------------------|-------------------|-----------------|-----------------|------------------|
| 0.055 | 0.76 | 0.01 | 0.001 | 0.27 | 0.17 |
| (0.001) | (0.01) | (0.0004) | (0.0005) | (0.01) | (0.01) |
| <u>Nickel</u> | Molybdenum | Copper | Tungsten | Vanadium | <u>Cobalt</u> |
| 36.1 | 0.01 | 0.08 | (0.04) | 0.02 | 0.06 |
| (0.1) | (0.005) | (0.005) | | (0.005) | (0.004) |
| <u>Tin</u> | <u>Aluminum</u> | <u>Niobium</u> | <u>Titanium</u> | Lead | Boron |
| (0.002) | (0.002) | (0.01) | (0.002) | | |
| | | | | | |
| <u>Nitrogen</u> | <u>Oxygen</u> | <u>Selenium</u> | Tantalum | <u>Zinc</u> | Zirconium |
| (0.003) | (0.01) | (0.17) | < 0.2 | | |

Iron of (62.2) is calculated by difference and is NOT CERTIFIED.

The laboratories participating in the "Interlaboratory Analysis Program" (ILAP) and certification of this material are as follows:

Anderson Laboratories Inc., Greendale, WI Armco Advanced Materials Co., Butler, PA Baltimore Specialty Steel Corp., Baltimore, MD Chicago Spectro Service Laboratory, Inc., Chicago, IL Earle M. Jorgensen Company, Seattle, WA Hoesch Stahl AG, Dortmund, Germany Metal Analysis Incorporated, Huntington Park, CA Shenango Industries, Inc., Terre Haute, IN Techni-Cast Corporation, South Gate, CA The Vollrath Company, Inc., Sheboygan, WI

Traceability to NIST: Members of the "Interlaboratory Analysis Program" (ILAP) listed above validate test methods and instrument performance utilizing SRMs produced by the National Institute of Standards & Technology, (NIST). Validation may be in the form of one or both of two independent methods described as follows:

- 1. Selected laboratories from the above listing are furnished SRMs that have been chosen as the best available "Type Standards" in relation to the matrix composition of the test materials being analyzed. These SRMs are provided by and at the expense of Analytical Reference Materials International, Inc. The data resulting from these examinations therefore becomes the common "point of reference" for all testing and subsequent data results for all IARMsTM certified by Analytical Reference Materials International, Inc. thereby establishing a specific line of "Traceability" to the U.S. Department of Commerce, National Institute of Standards & Technology, (NIST). The specific SRMs applicable to the material covered by this certificate were SRM1155, SRM1219, SRM1267, and SRM1230.
- 2. At the option of ARMI and/or in the absence of currently available SRMs that are matrix compatible with ARMI program materials, other NIST/NBS materials classically resident in the ILAP member lab may be used for the characterization of ILAP materials. Validation by this method requires that the ILAP member have on file with ARMI an affidavit verifying the use of the SRMs accompanied by a copy of the NIST/NBS Certificates of Analysis as they apply to that matrix group.

Either one or both of these methods establishes a specific line of traceability to NIST for those elements that are noted as "certified values" on the NIST Certificates of Analysis applicable to the SRMs referenced on the ARMI Certificate of Analyses.

See Reverse Side for Statistical Data and Additional Information Regarding This Material

The following data and accompanying statements represent all pertinent information reported in the interlaboratory program as it applies to the chemical characterization of this material as of 06/22/1994.

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| Lab | Fe | С | Mn | Р | S | Si | Cr | Ni | Мо | Cu | W | V | Со |
|-----------|--------|--------|--------|--------|--------|--------|--------|-------|--------|--------|--------|--------|--------|
| 1 | | 0.056 | 0.7734 | 0.011 | 0.002 | 0.28 | 0.18 | 36.27 | 0.02 | 0.08 | 0.046 | 0.03 | 0.06 |
| 2 | | 0.0559 | 0.77 | 0.01 | 0.002 | 0.28 | 0.18 | 36.18 | 0.01 | 0.08 | 0.04 | 0.03 | 0.06 |
| 3 | | 0.055 | 0.77 | 0.01 | 0.002 | 0.27 | 0.1722 | 36.11 | 0.01 | 0.08 | 0.03 | 0.025 | 0.057 |
| 4 | | 0.055 | 0.77 | 0.01 | 0.0017 | 0.27 | 0.17 | 36.00 | 0.01 | 0.08 | 0.025 | 0.02 | 0.056 |
| 5 | | 0.055 | 0.761 | 0.01 | 0.001 | 0.26 | 0.17 | 35.95 | 0.006 | 0.072 | | 0.02 | 0.055 |
| 6 | | 0.054 | 0.76 | | 0.001 | 0.25 | 0.165 | 35.91 | | 0.07 | | 0.019 | 0.05 |
| 7 | | | 0.75 | | 0.001 | 0.246 | 0.16 | | | | | | |
| 8 | | | | | 0.001 | | 0.16 | | | | | | |
| 9 | | | | | | | 0.16 | | | | | | |
| | | | | | | | | | | | | | |
| M/A | | 0.0552 | 0.7649 | 0.0102 | 0.0015 | 0.2651 | 0.1686 | 36.07 | 0.0112 | 0.0770 | 0.0353 | 0.0240 | 0.0563 |
| STDV. | | 0.0007 | 0.0083 | 0.0004 | 0.0005 | 0.0136 | 0.0080 | 0.14 | 0.0052 | 0.0047 | 0.0095 | 0.0051 | 0.0037 |
| Certified | (62.2) | 0.055 | 0.76 | 0.01 | 0.001 | 0.27 | 0.17 | 36.1 | 0.01 | 0.08 | (0.04) | 0.02 | 0.06 |

Continued

| Lab | Sn | Al | Nb | Ti | Pb | В | N | 0 | Se | Та |
|-----------|---------|---------|--------|---------|---------|---|---------|--------|--------|--------|
| 1 | 0.002 | 0.003 | 0.01 | 0.002 | <0.0001 | | 0.0038 | 0.0115 | 0.17 | 0.18 |
| 2 | 0.002 | 0.001 | 0.01 | 0.002 | | | 0.003 | 0.007 | 0.16 | |
| 3 | 0.001 | 0.001 | 0.01 | 0.001 | | | 0.003 | | | |
| 4 | | 0.001 | <0.01 | 0.001 | | | 0.0024 | | | |
| 5 | | | | <0.005 | | | 0.0021 | | | |
| 6 | | | | | | | 0.001 | | | |
| | | | | | | | | | | |
| M/A | 0.0017 | 0.0015 | 0.01 | 0.0015 | | | 0.0026 | 0.0093 | 0.1650 | 0.1800 |
| STDV. | 0.0006 | 0.0010 | | 0.0006 | | | 0.0010 | 0.0032 | 0.0071 | |
| Certified | (0.002) | (0.002) | (0.01) | (0.002) | | | (0.003) | (0.01) | (0.17) | <0.2 |

The International Standards Organization (ISO) definitions, expressed in ISO Guide 30-1981- (E) list the following:

Certifying Body: A technically competent body (organization or firm, public or private) that issues a Reference Material Certificate. The only generally accepted certifying body in the United States is the U. S. Department of Commerce, National Institute of Standards & Technology, (NIST), Gaithersburg, MD.

Reference Material (RM): A material or substance with one or more properties which are sufficiently well established to be used for calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Certified Reference Material (CRM): A reference material with one or more properties whose values are certified by a technically valid procedure accompanied by or traceable to a certificate or other documentation, which is issued by a Certifying Body.

Interlaboratory Analysis Program: Although ASTM Standard E691-87 applies to interlaboratory studies to "Determine the Precision of a Single Test Method", it is also a well thought out and logical plan for conducting an interlaboratory program involving multiple techniques. Therefore, the planning, conducting, analyzing, protocol and treatment of data resulting from this interlaboratory program were performed utilizing the guidelines established in ASTM E691-87.

Methods of Analysis: In view of the fact that the "Interlaboratory Analysis Program" entails a wide variety of materials, no single analytical method would provide optimum data results. Therefore the methods utilized were a combination of ASTM Standard Methods for classical wet chemistry, ICP, AA, Optical Emission and X-Ray spectrometric methods. The determinations for Carbon, Sulfur, Nitrogen and Oxygen are the result of combustion instrument procedures.

Selection of Materials: A "batch" or "series" is defined as a single bar of one continuous length. The majority of materials are in wrought condition. Other methods of manufacture are utilized as a last resort, only in the case of those materials being unavailable in wrought condition. "Batch" samples are taken by removing a one inch cross section for every thirteen inches of total length from the entire bar. Twenty-five percent of the one inch cross section is converted to chips for analysis by classical wet chemistry, ICP, AA, and combustion procedures and seventy-five percent remains in a solid disk form for OES and X-Ray analysis where applicable. Each member of the ILAP is furnished both a solid sample and the corresponding supply of chips from a specific location on the batch bar. This massive sampling procedure results in the homogeneity being reflected as a product of the overall statistics and certified data. To insure that there are no gross abnormalities in the batch homogeneity, effective with materials certified after 10/20/91, specific homogeneity testing is being performed utilizing applicable sections, determined by the ILAP program management, for homogeneity test procedures as outlined in ASTM E-826-87, NBS Handbook 91, "Experimental Statistics", as well as other established practices for determination of practical homogeneity.

Certified by

R. Dan Brown, President

Analytical Reference Materials International

Certificate No.: 24A-021591ARM-F Certification Date: 02/15/1991

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